UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

REPLY TO THE ATTENTION OF:
5SMQA

MEMORANDUM

DATE: JUN 28 1989

SUBJECT: Approval of the First Revision, Fund-Lead Quality Assurance Project Plan for the Sampling and Analysis of Fishes - as Part of the Remedial Design Activity at the Republic Steel Quarry Superfund Site in Elyria, Ohio

FROM: Valerie J. Jones, Chief
Monitoring and Quality Assurance Branch

TO: Norman Niedergang, Chief
Remedial and Emergency Response Branch

ATTENTION: Kenneth Tindall, RPM

The Quality Assurance Section (QAS) is providing approval of the first revision, Fund-Lead Quality Assurance Project Plan (QAPjP) for the sampling and analysis of fishes (QAS Log-In No. 960) - as part of the Remedial Design (RD) activities at the Republic Steel Quarry Superfund site in Elyria, Ohio, with the following changes:

1. In Section 10.1 (page 21 of 27), a sentence, "Data reporting format will be consistent with the CLP Statement of Work, SOW-7/88 for inorganic analysis, except the analysis results will be reported on the basis of wet weight."

2. In the Special Analytical Service (SAS) request, changes are made as follows:

   a. The name of Regional Representative and the telephone number are changed to "Jan Pels" and "353-2720" respectively.

   b. The referenced document number of the CLP Statement of Work in Item #1 and #7 are changed from "8/87" to "7/88".

   c. In Item #1, the text is revised to include the determination of % lipids as part of the required analysis.

   d. In part 6 of Item #8, the text is revised to read, "Digested method blank and reagent blank will be prepared at a frequency of one per 10 samples digested, or one per batch if a batch is less than 10 samples; and will be analyzed at the beginning of each day before the analysis of any samples, after every 5 samples analyzed, and at the end of daily analysis."

e. At the end of part 7,Item #8, "See also Item #10 for matrix spike." is added.

f. In Item #9, the text is revised to read, "As per standard CLP reporting package, except the analytical results will be reported on the basis of wet weight."

g. The acceptance control limits for duplicate analysis and matrix spike analysis are changed to "+/- 35% RPD" and "75-125% recovery" respectively.

h. In Item #13, the frequency of audit for method blank is revised to read,"See part 6 of Item #8 for frequency of preparation and analysis of method blank."

A copy of these corrected pages is included for your use, and they shall be incorporated in the finalized copy of the QAPjP.

The original signature page is included. Please have the Remedial Project Manager provide final sign-off. We have retained two copies of this subject QAPjP for the Central Regional Laboratory's and our records. We would like to receive a copy of the completed signature page when it is available.

Attachment

cc: Cheng-Wen Tsai, ESD/QAS

10.0 DATA REDUCTION, VALIDATION AND REPORTING

10.1 CLP-SAS

The test procedures used by SAS will be clearly identified. Bench records and all records of analyses and calculations for samples, blanks, duplicates, spikes, and standards, with resultant instrument inputs or concentration readouts, will be provided by CLP, SAS, along with worksheets used to calculate results. The laboratory analytical data will be reported in accordance with standard CLP protocol as specified in the SAS Regional Request Form (Appendix B). Data validation will be performed by the Laboratory Scientific Support Section (LSSS) and reviewed by ICF personnel. Data validation will be performed in accordance with the standard EPA Region V validation procedures, which are based on the EPA National Functional Guidelines for Evaluating Inorganic Analyses. The raw data collected from project sampling tasks and used in project reports will be identified and will be included in a separate appendix within the final report. Where test data have been reduced, the method of reduction will be described.

Data reporting format will be consistent with the CLP Statement of Work, SOW-7/88 for inorganic analysis except the results will be reported on the basis of wet weight.

10.2 Field Analysis

All field recording sheets, instrument outputs, and worksheets for calculating results will be retained. Summarized raw data will be appropriately identified in reports and included in a separate appendix in the final RI report. Where test data have been reduced, the method of reduction will be described.

.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: EPA Region 5/Republic Steel Quarry

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: June 9, 1989

E. Site Name: Republic Steel Quarry

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Fish tissue analysis for mercury, manganese, and % Lipid. The tissues will be homogenized and frozen prior to submittal to the lab. The lab must digest the samples using the attached nitric acid/perchloric acid method, after which the digestate will be analyzed using the 7/88 CLP inorganic SOW.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

A total of 19 fish tissue samples will be sent for analysis, including two blanks (obtained from a clean source), two duplicates, and two spikes (prepared during homogenization).

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):

Superfund - Remedial Action

4. Estimated date(s) of collection: August, 1989

5. Estimated date(s) and method of shipment: August, 1989 via Federal Express

6. Approximate number of days results required after lab receipt of samples:

30 days

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Digest the tissue as detailed in Section 11 of the attached procedure, "Analysis of Metals and Metalloids in Estuarine and Marine Tissues." Analyze the digestate using the 7/88 inorganic SOW, omitting the digestion step.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
Note: The fish will be filleted after which the skin-on fillet will be homogenized, frozen, and sent to the lab. The lab is to analyze the samples as received, and report the results on a wet weight basis.

1. Follow Sections 1.0, 2.0, 3.0, 4.0, 5.0, 6.4-6.6, 7.0, 10.6, 11.3-11.7 and 12.0 of the attached procedure.
2. The samples must be stored in the freezer (0°F) from time of receipt until analysis.
3. The procedure makes use of glass reflux caps (Tuttle caps). Water cooled reflux condensers must be used in place of the caps.
4. The mercury and manganese standards should be made up to have the same final concentration of nitric acid and perchloric acids as the samples.

5. Analyze the digestate for mercury using inorganic SOW Exhibit D, Section IV, Part D; for manganese using inorganic SOW Exhibit D, Section IV, Part A; and for percent solids using inorganic SOW Exhibit D, Section IV, Part F.

6. Digested method blanks and digested reagent blanks will be prepared at a frequency of one per 10 samples digested, or one per batch if a batch is less than 10 samples; and will be analyzed at the beginning of each day before the analysis of any samples, after every 5 samples analyzed, and at the end of the daily analysis.

7. All of the QA/QC procedures described in the inorganic SOW Exhibit E should be followed. This includes instrument calibration, initial calibration verification, continuing calibration verification, CRDL standard for ICP, initial calibration blank, continuing calibration blank, preparation blank, ICP interference check, spike sample, duplicate sample, laboratory control sample, ICP serial dilution, and ICP inter-element corrections. See also Item #10 for matrix spike.

8. Determine the percent lipids using the attached procedure.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

As per standard CLP reporting package, except analytical results will be reported on the basis of wet weight.

10. Other (use additional sheets or attach supplementary information, as needed):

Spike samples will be prepared by the lab. The spike added will increase the concentration of mercury and manganese by a factor of three (3) times the detection limit. The spiking solutions will use methyl mercuric chloride and manganese nitrate as the solutes. The spike samples must be prepared prior to sample digestion. The samples must be homogenized for at least 10 minutes using a blender following addition of the spike.

11. Name of sampling/shipping contact: Paul Tomiczek

Phone: (412) 788-9200

12. **Data Requirements**

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Mercury	20 ug/kg	± 25%
Manganese	100 ug/kg	± 25%

13. **Quality Control Requirements:**

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Method Blank	Per part 6 of Item #8 for frequency of praparation and analysis of method blank	<Detection Limit
Duplicate (as per Exhibit E Section 7)	1 per 10 samples	±35%RPD
Spike (as per Exhibit E Section 6)	1 per 10 samples	75-125% Recovery

14. **Action Required if Limits are Exceeded:**

1. Take corrective action and re-analyze the samples.

2. Contact Jay Thakker (312-886-1972) or Chuck Elly (312-353-9087)

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

PROJECT TITLE: Republic Steel Quarry, Elyria, Ohio

EPA No.: 84-5LW6.0

EPA PROJECT OFFICER: Kenneth W. Tindall

PREPARED BY: ICF Technology Inc. Date:_____

Approved _____ Date:_____
CH2M Hill Review Team Leader

Approved _____ Date:_____
ICF Site Project Manager

Approved _____ Date:_____
EPA Remedial Site Project Officer

Reviewed _____ Date:_____
Director, Central Regional
Laboratory, EPA Region V

Approved Date: 6/26/89
EPA QA Officer

(REM IV) ZONE II
Contract Number 68-01-7251
REPUBLIC STEEL QUARRY
ELYRIA, OHIO
QUALITY ASSURANCE PROJECT PLAN (QAPP)

PROJECT TITLE: Republic Steel Quarry, Elyria, Ohio

EPA No.: 84-5LW6.0

EPA PROJECT OFFICER: Kenneth W. Tindall

PREPARED BY: ICF Technology Inc. Date:_____

Approved _____ Date:_____
CH2M Hill Review Team Leader

Approved _____ Date:_____
ICF Site Project Manager

Approved _____ Date:_____
EPA Remedial Site Project Officer

Reviewed _____ Date: 6/29/89
Director, Central Regional
Laboratory, EPA Region V

Approved _____ Date: 6/26/89
EPA QA Officer

TABLE OF CONTENTS

2.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) for the Republic Steel Quarry Site presents specific procedures adopted by CH2M Hill and ICF Technology (ICF) to fulfill the requirements of EPA's Quality Assurance (QA) program. This QAPP addresses:

- The QA objectives of the project.
- Specific QA and QC (quality control) procedures that will be implemented to achieve these objectives.
- Staff organization and responsibility.

The requirements of EPA with regard to QA focuses on the acquisition of environmental data of known and acceptable quality. Other aspects of the project, such as engineering analysis and report preparation, will be controlled by internal requirements of Quality Assurance Programs for CH2M Hill and ICF.

3.0 PROJECT DESCRIPTION

3.1 Objectives

The primary objective of this field investigation is to collect data to establish or refute the existence of public health hazards due to ingestion of fish from the Republic Steel Quarry site. The Endangerment Assessment (EA) performed as part of the Phase I Remedial Investigation (RI) concluded that ingestion of fish from the quarry was the only current-use exposure scenario resulting in greater than 10^{-6} excess lifetime cancer risk. This maximum exposure scenario also resulted in a hazard index greater than one due primarily to manganese and mercury. Because the risks were estimated using conservative contaminant uptake models and not actual fish samples, fish sampling and analysis will be performed to confirm or refute the presence of the risks due to fish ingestion.

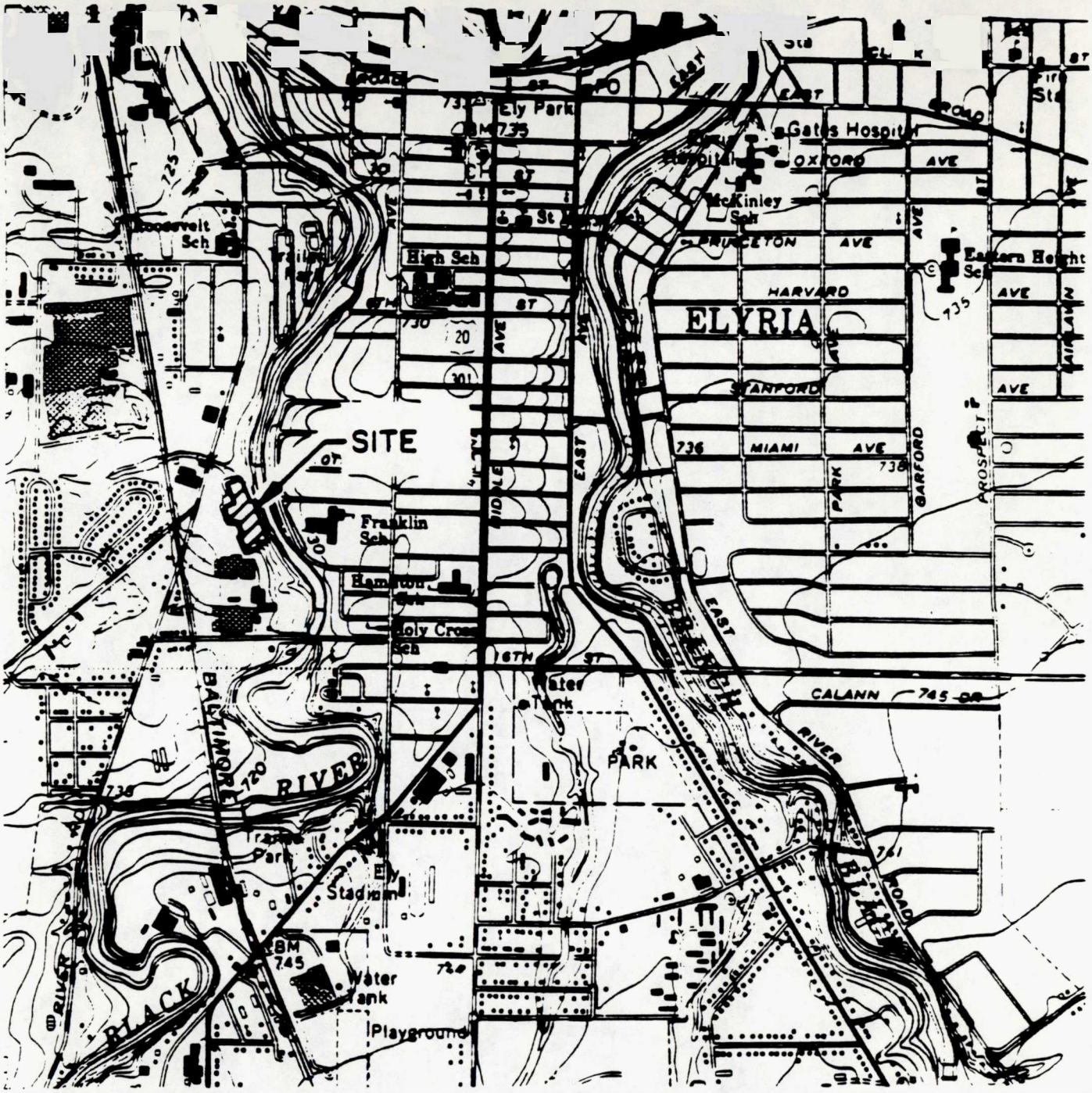
3.2 Site Description

Site Description: The Republic Steel Quarry site is located east of West River Road and west of the West Branch of the Black River, directly across the river from Franklin School in Elyria, Ohio (see Figure 1). The City of Elyria is located southwest of Cleveland in Lorain County in northeastern Ohio. The site can be found on the Grafton USGS quadrangle map in Township 6 North, Range 17 West.

The site consists of a five acre quarry and the fenced area surrounding the quarry (see Figure 2). Water in the quarry has been measured at depths up to 62 ft. The sides of the quarry are nearly vertical and rise to an average of about 20 to 30 ft. above the quarry water surface elevation. The quarry walls are composed of in-place Berea Sandstone at and below the present water level. Above the sandstone, the quarry walls are composed of vertically stacked, large sandstone blocks. These blocks were placed during quarry operations as retaining walls to support the soil zone.

Although the site is fenced, it is still accessible through holes in the fence and areas where the bottom of the fence is 1 to 3 ft. above the ground surface. Water from the quarry discharges directly to the West Branch of the Black River. Water in the quarry is in direct contact with the Berea Sandstone formation which is a water supply aquifer in the area. Vegetation around the quarry perimeter is mostly grass and small brush; however, several larger trees can be found around the site and along the river. Vegetation is fairly dense over most of the site.

There are two hydraulic systems in the quarry. Concrete outlet works equipped with a gate valve are located along the east quarry wall where the elevation dips to about 704 ft. mean sea level (MSL). Water is usually draining from the outlet works into the river.



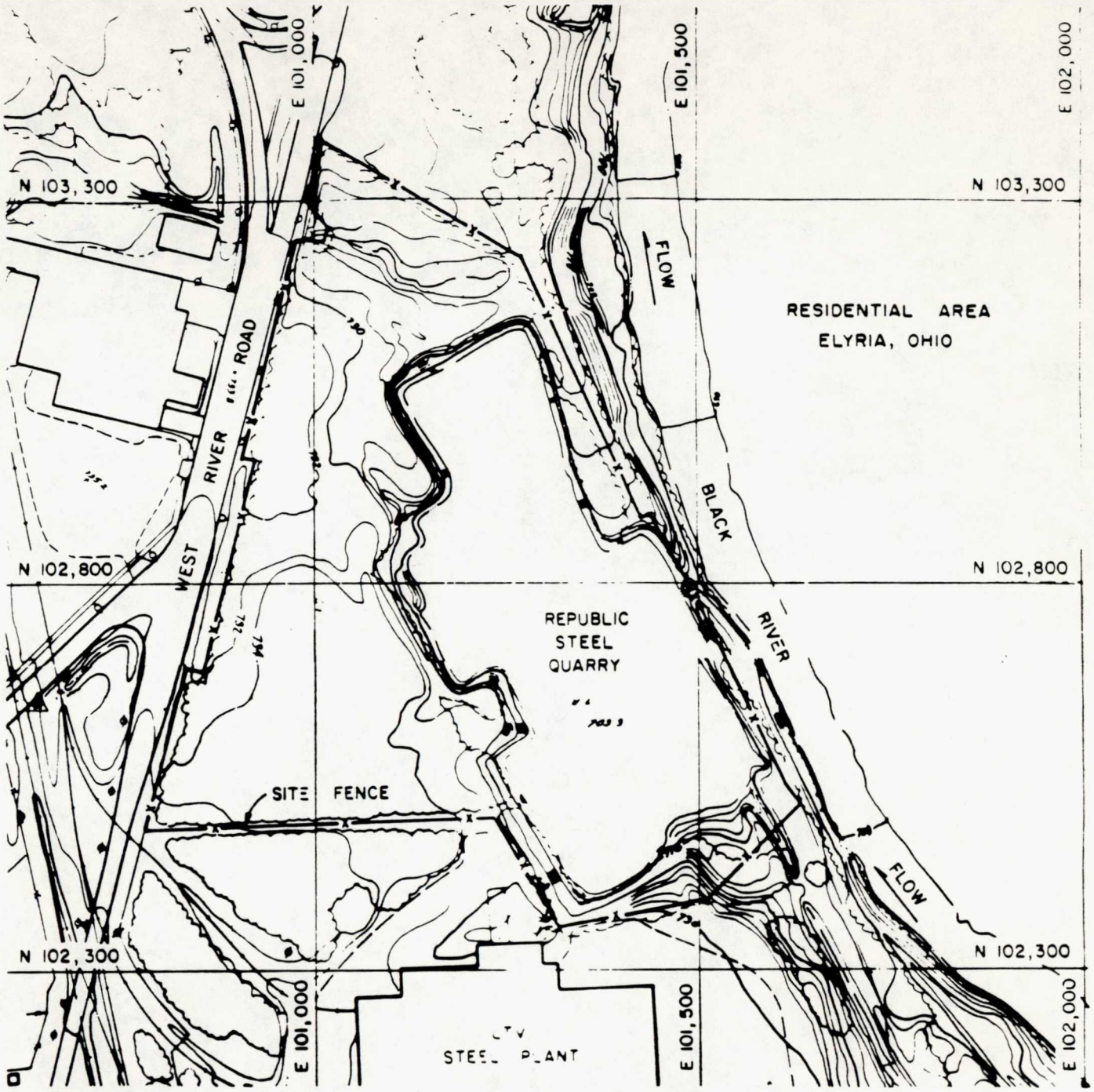
SOURCE: U.S.G.S. 7.5' TOPOGRAPHIC MAP GRAFTON QUADRANGLE OHIO, SCALE: 1" = 2000'.

SITE LOCATION

0 1540 3080

SCALE IN FEET

FIGURE 1
SITE LOCATION MAP
REPUBLIC STEEL QUARRY SITE



SOURCE: CITY OF ELYRIA
ENGINEERING DEPARTMENT
SCALE: 1" = 100'

N

0 200 400

SCALE IN FEET

FIGURE 2
SITE MAP
REPUBLIC STEEL QUARRY SITE

A 4 in. diameter steel pipe is located at the southeastern-most corner of the quarry extending down into the water. This pipe is believed to have been used for withdrawing water from the quarry to the Republic Steel plant, rather than being used for discharging the pickle liquor wastes. Neither system is apparently operational.

LTV Steel acquired Republic Steel Corporation, and is currently operating the steel plant located southwest of the quarry. There is an abandoned municipal landfill located about 0.6 miles northwest of the site. EPA closed this landfill several years ago. Land surrounding this landfill was flat, and this landfill was observed to have only a very slight mounding of the ground surface. Because of the surrounding land topography, it is thought that the wastes may have been disposed in a pit or quarry rather than in an above-ground landfill. There is also an industrial complex located 0.6 miles northwest of the site.

3.3 Site Background

The Republic Steel Quarry site was operated as a sandstone quarry during an unknown period of time prior to 1950. Stone cut from the quarry was the Berea Sandstone, which was used as a building material.

From 1950 to 1972 Republic Steel Corporation discharged waste pickle liquor to the five acre quarry. The approximate volume of waste disposed into the quarry was estimated to be 200,000 gallons/year by Republic Steel personnel. The quarry water was sampled by Republic Steel Corporation in March and April of 1976. Stratified water quality was detected with the deeper waters having higher iron concentration and lower pH than shallower waters. The pH of surface samples was approximately 7, while the pH of the quarry bottom samples was about 2. In 1977, the quarry was sold to the City of Elyria.

In 1981, Republic Steel Corporation notified EPA of the disposal activity under Section 103(C) of the Comprehensive Environmental Responsibility, Compensation, and Liability Act (CERCLA). In response to Republic Steel's notification, Ecology and Environment, Inc. (E & E) performed a site investigation for EPA as its Field Investigation Team (FIT) contractor in late 1983 and installed three monitoring wells. Water samples were collected from the quarry and the monitoring wells. No organic contamination was detected at the site; however, heavy metals such as chromium, arsenic, lead, cadmium, magnesium, aluminum and iron were detected at significantly higher levels in the groundwater at one downgradient monitoring well than in the upgradient wells. The quarry water samples were collected from the surface and at depths of 10 ft., and only iron was detected at high concentrations.

The site was evaluated relative to the Hazard Ranking System in 1984. On October 15, 1984, EPA proposed that Republic Steel Quarry be included in Group II of the National Priorities List (NPL). IT Corporation (IT), at the request of LTV Steel, conducted an investigation of the site in November, 1984. Groundwater samples collected during this investigation detected higher concentrations of inorganics in the upgradient wells than in the downgradient

wells. The quarry water samples collected had nearly neutral pH values (6.2 to 6.5), but were collected from only the surface and depths of 10 ft. This investigation concluded that EPA's Hazard Ranking System score should be recomputed based on evidence obtained in IT's investigation. This recomputed score, according to IT, would not be high enough for the site to be included on the NPL.

The results of the previously performed investigations were conflicting. The original investigation performed by Republic Steel detected potential hazards associated with the quarry water. The groundwater data collected by the FIT contractor concluded that groundwater contamination was probably occurring, while the PRP contractor's data concluded that no problem existed. Inconsistent groundwater sampling techniques could be the cause of the differences.

A remedial investigation was conducted by ICF Technology to confirm or refute the presence of site related environmental and public health hazards. A summary of the Phase I RI activities and findings is presented in Section 3.4.

3.4 Summary of Phase I RI Activities

The field activities of the RI were performed from June through August, 1987. The major activities utilized to collect data during the RI included:

- Profiling of quarry water quality and depth;
- Performance of a magnetometer survey to identify metal objects on the quarry bottom;
- Reconnaissance of quarry surface geology;
- Chemical characterization of surface water from the quarry and the Black River;
- Chemical characterization of sediment from the quarry and the Black River;
- Chemical characterization of surface soils adjacent to the quarry;
- Installation of eight monitoring wells and sampling of groundwater;
- Property boundary research.

3.4.1 Major Findings

Samples of surface water, sediments, surface soils and groundwater were collected and analyzed to estimate the types and extent of contamination due to the site. The conclusions with respect to the contamination of each media are included in the following items.

Surface Water: Surface water samples were collected from the quarry and Black River adjacent to the site during June 1987. Resampling was performed during March 1988 for semi-volatile organic analyses due to laboratory quality control problems that occurred during the initial analyses. To estimate the extent and nature of contamination, two analyses of the results were performed. Initially, the quarry water samples were compared to upgradient groundwater samples to identify potentially site related chemicals in the quarry. No organic chemicals were identified in the quarry water as being potentially site related. Several inorganic chemicals were identified as being potentially site related. All of these chemicals were detected at elevated concentrations compared to upgradient groundwater, with the greatest concentrations observed near the quarry bottom. The downstream Black River samples were compared to upstream river samples and quarry samples to estimate if the site was affecting river water quality. The results of this comparison indicated that the site was not adversely impacting Black River water quality.

Sediments: Sediment samples from the quarry and the Black River were collected in June 1987 and analyzed for organic and inorganic chemicals. The nature and extent evaluation for sediments was performed in two phases to determine if the quarry sediments were contaminated and to estimate if the quarry had adversely impacted the quality of Black River sediments. Initially, quarry sediments were compared to background soils to identify potentially site related chemicals. This analysis concluded that sediments within the quarry contain elevated levels of volatile and semi-volatile organic chemicals and inorganic chemicals. Volatile organic compounds were detected only in the deep quarry sediment samples (greater than 35 ft.) while semi-volatile organics and inorganics were detected in both deep and shallow samples. Concentrations of the inorganic and semi-volatile contaminants of the sediments obtained from deeper portions of the quarry were greater than those from the shallow sediments. The evaluation indicates that past activities at the site have affected quarry sediment quality.

A comparison of downstream Black River sediments to upstream river and quarry sediments was performed to estimate if the site is adversely impacting Black River sediments. The analysis indicated that no potentially site related organic or inorganic chemicals were detected in the sediments downstream. The site is not affecting sediments in the Black River.

Surface Soils: Surface soil samples were collected in June 1987. Analyses performed on surface soils obtained from areas of the site that are periodically inundated by quarry water or that were exposed to waste discharges in the past detected contaminants above background concentrations. Contaminants detected included volatile and semi-volatile organic chemicals and inorganic chemicals. Past disposal activities appear to have affected the quality of surface soils at the site. Semi-volatile organic chemicals and inorganic chemicals were also detected in a sample of the steel yard soils that are sliding into the quarry.

Groundwater: Eight monitoring wells were installed at or near the Republic Steel Quarry Site. All of these wells were sampled in August 1987 and two wells were resampled and analyzed for volatile organics in March 1988 and October 1988. Based on the two resampling efforts, groundwater at the site is not contaminated with organic compounds. Inorganic chemicals were detected in all downgradient wells adjacent to the site and in the well across the Black River; however, a direct connection to the site cannot be made to the inorganic chemicals in well B-8. Moreover, it is unlikely that contaminants from the quarry are passing beneath the Black River within groundwater since well B-8 is hydraulically upgradient from the site.

3.4.2 Endangerment Assessment Summary

The potential risks to human and environmental health attributed to chemicals present at the Republic Steel Quarry Site were evaluated in the Endangerment Assessment under a number of exposure scenarios. Potential pathways of exposure to chemicals originating at the site under both current-use and hypothetical future-use conditions were examined.

Under current-use conditions, the only exposure scenario resulting in a greater than 10^{-6} risk is the maximum case for ingestion of fish. This risk is primarily due to carcinogenic PNAs which were conservatively estimated using the sediment/surface water exposure model. Additionally, this maximum exposure scenario results in a total noncarcinogenic hazard index greater than one due primarily to mercury and manganese. This risk is based on modeling using very conservative assumptions. Combined risks to trespassers, assuming the same person would be exposed to soil through direct contact and incidental ingestion, quarry water through swimming, and fish through ingestion were estimated. The combined upperbound excess lifetime cancer risks are 3×10^{-8} to 4×10^{-6} under average and maximum exposure conditions. The combined hazard index is less than one under the average scenario and greater than one under the maximum scenario.

Based on conversations with personnel from the National Fisheries Contaminant Research Center, U. S. Fish and Wildlife Service, and National Oceanic and Atmospheric Administration (NOAA), and a review of published literature, USEPA, Ohio EPA and ICF concluded that detectable levels of PNAs will not be present in fish tissue at the Republic Steel Quarry site because PNAs will be metabolized by the fish. Because the PNAs are metabolized by the fish and detectable concentrations of PNAs in fish tissue samples are not expected, ingestion of fish tissue will not cause unacceptable carcinogenic health risks. No other organic compounds were determined to pose a significant health risk in the Endangerment Assessment; therefore, USEPA, Ohio EPA and ICF concluded that manganese and mercury concentrations in fish tissue should be evaluated to estimate the noncarcinogenic health hazards posed by fish tissue ingestion.

3.5 Scope

The field data gathering efforts planned for the investigation include two tasks. These tasks include:

- Fish Species Survey - This task will involve collecting and identifying fish throughout the quarry and Black River adjacent to the site so that species composition and relative abundance will be established. Feeding habits will be determined based on published reference data rather than field survey data. Fish will be identified to the species level. Fish length and weight will be measured.

- Fish Tissue Sampling - Tissue samples will be prepared by processing several specimens per individual sample (if available), for inorganic laboratory analysis. Large and small fish fillet samples for certain species are proposed. A total of seventeen (17) fish tissue samples, including blanks and duplicates, are planned. Proposed samples are presented on Table A-1 in the Sampling Plan in Appendix A.

4.0 PROJECT ORGANIZATION AND RESPONSIBILITY

CH2M Hill has overall responsibility for all phases of the RI/FS. ICF Technology is a REM IV Associate Firm to CH2M Hill. ICF will perform the field investigations and prepare the RI report as part of the Remedial Investigation and will prepare the Feasibility Study. Both ICF and CH2M Hill will provide project management as appropriate to their responsibilities.

The following responsibilities have been assigned for the project:

- Remedial Project Manager (RPM)
  Kenneth Tindall (EPA)

- Site Manager (SM)
  Kenneth Miller (ICF)

- Quality Assurance Manager (QAM)
  Greg Peterson (CH2M Hill)

- Review Team Leader (RTL)
  Andrew Diefendorf (CH2M Hill)

- Field Operations
  Paul Tomiczek (ICF)

- Sample Team Leader
  Patrick Sullivan (ICF)

- Laboratory Operation for Routine and Special Analytical Services
  Contract Laboratory Program (CLP)

- System/Performance Audits , CH2M Hill QA Manager and ICF Field Operations Coordinator (field analyses), U. S. EPA EMSL-Las Vegas (CLP)

- Special Analytical Services Request Preparation
  Richard McCracken (ICF)

- Review of Tentatively Identified Compounds
  Richard McCracken (ICF)

- QA/QC of CLP Data - Sample Management Office (SMO) and U. S. EPA Region V Laboratory Scientific Support Section (LSSS)

- CLP Data Completeness
  Richard McCracken (ICF)

- Site Safety Officer
  Daniel Welshons (ICF)

EPA's Remedial Project Manager is responsible for overseeing the entire project. The QA Officer is responsible for review and approval of the Quality Assurance Project Plan. Primary responsibility for project quality rests with the SM. Independent quality assurance review is provided by the QA reviewers. A project organization chart is presented on Figure 3. The proposed project schedule is presented on Figure 4.

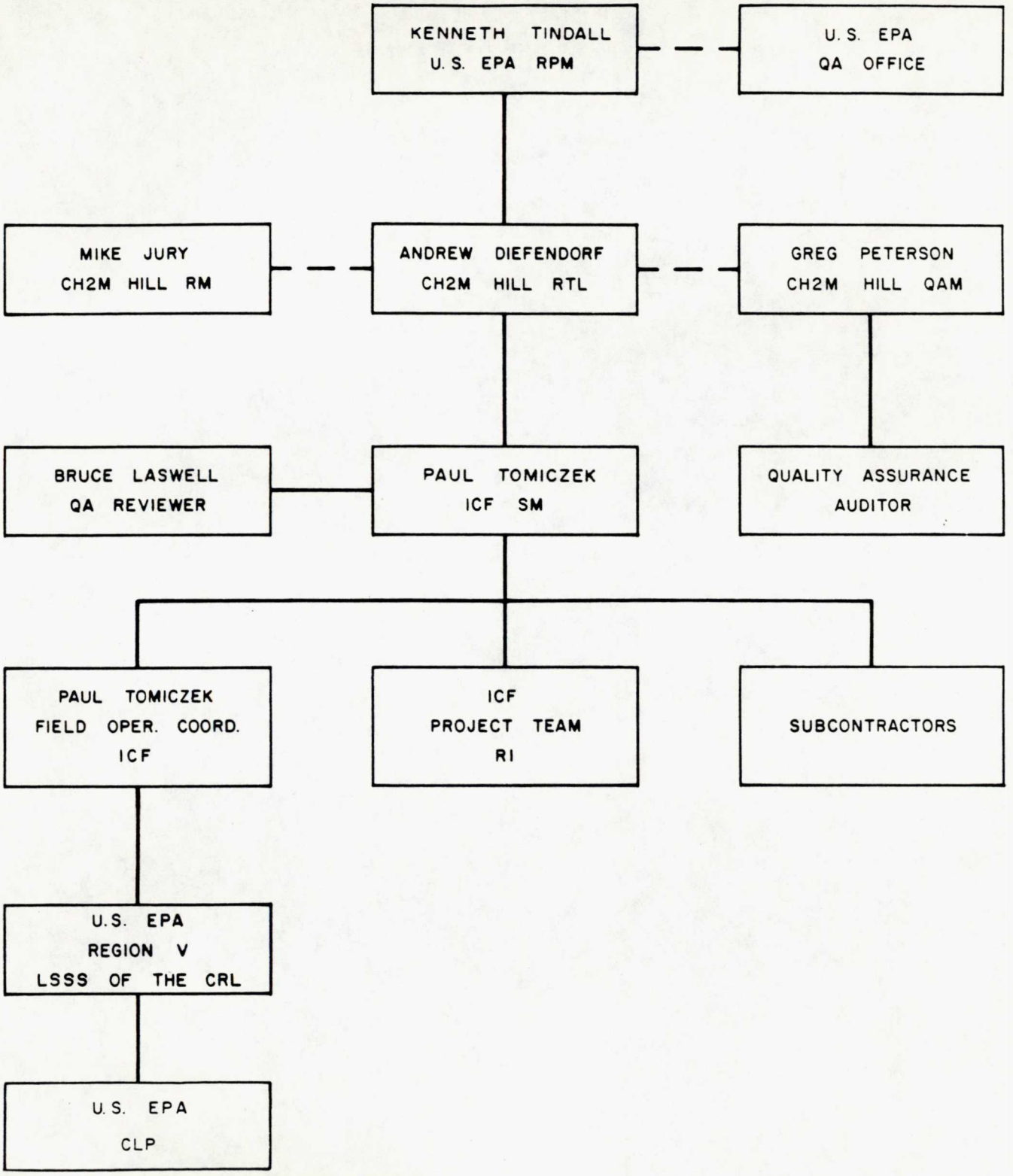
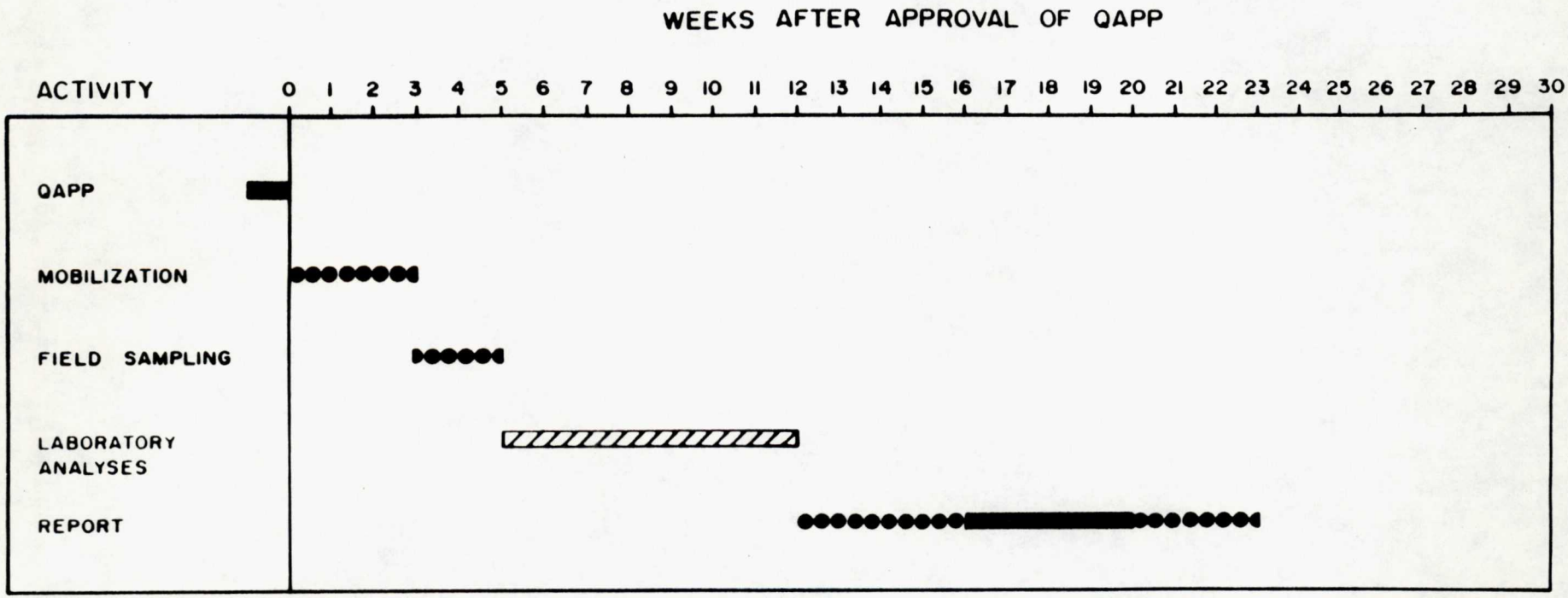


FIGURE 3
PROJECT ORGANIZATION
REPUBLIC STEEL QUARRY SITE



LEGEND:

AGENCY REVIEW

CONTRACTOR'S ACTIVITY

LABORATORY ANALYSIS

FIGURE 4
PROPOSED SCHEDULE
REPUBLIC STEEL QUARRY SITE

5.0 QUALITY ASSURANCE OBJECTIVES

5.1 Data Quality Objectives

For each of the data-gathering tasks listed in Section 3.5, Data Quality Objectives (DQOs) have been developed. These DQOs present the intermediate and end uses of the generated data and the resultant data quality which is required.

5.1.1 Fish Species Survey

The purpose of the fish species survey is to provide data on the types of fish that inhabit the quarry and Black River adjacent to the site. The information gained from the fish species survey will establish species composition and relative abundance. The species data will allow an evaluation of the number and types of fish that may be ingested by persons fishing in the quarry. Additionally, identifying the types of smaller fish that the larger fish are feeding on will be useful in estimating contaminant uptake and bioaccumulation through the food chain. Fish length and weight measurements will be obtained.

All fish will be identified to the species level by trained personnel, familiar with fish identification. Reference materials will be maintained on site for use as required. The representativeness and comparability of the data will be ensured by cross-referencing the fish species identified with published reference materials, including Trautman's Fishes of Ohio. Data will be comparable and accurate. One whole fish of each species identified in the quarry will be retained and stored in a 10% formaldehyde solution for at least a two year period to allow fish species confirmation if it becomes necessary at a later date during the project.

5.1.2 Fish Tissue Sampling

The purpose of the fish tissue sampling is to evaluate the presence or absence of contaminants within fish tissue for use in determining potential for adverse health effects due to long-term ingestion of fish from the quarry.

Samples of fish from each species identified will be selected and composited, and prepared for inorganic laboratory analyses. Representative fish from each species will be selected, rather than only fish appearing healthy, or only fish showing evidence of disease. The team will select a representative set of fish based on all fish from that species collected.

In all cases, several fish will be composited for one sample. Fish fillets with the skins on will be obtained for the composite sample. The compositing of several fish into one sample will ensure that a representative sample is obtained, eliminating the possibility of collecting one fish sample that is either much cleaner or more contaminated than the average. The data will be comparable and accurate through the quality assurance procedures to be utilized during fish collection, sample compositing, sample preparation, and decontamination. These procedures are described in detail in the sampling

plan presented as Appendix A. Data precision and accuracy requirements are specified in Section 14.1 of this QAPP. Samples of fish tissue from the Black River will be collected from above the dam and below the site to evaluate the risks of selected chemicals posed by ingestion of Black River fish. These data will be used for comparative purposes with quarry fish tissue.

6.0 SAMPLING PROCEDURES

Detailed sampling procedures are provided in the Sampling Plan, Appendix A.

7.0 SAMPLE CUSTODY

Sample custody procedures for this project will be in accordance with the procedures detailed in "NEIC Policies and Procedures," EPA-330/9-78-001-R, revised June 1985. These procedures divide custody into three areas: sample collection, laboratory, and final evidence files. Each of these is addressed in this section.

A sample or evidence file is under your custody if it:

- is in your possession;
- is in your view, after being in your possession;
- was in your possession and you placed it in a secure location; or
- is in a designated secure area.

7.1 Field Custody Procedures

The procedure outlined below will document sample custody from collection until arrival at the laboratory:

1. The field sampler has custody of the samples from the time they are collected until they are transferred to the sample packager.

2. All bottles are tagged with sample number, SAS number, and location by the sampler and/or packager. Sample tags are completed for each sample using waterproof ink, unless prohibited by weather conditions. Each bottle has a custody seal placed around its cap.

3. The sample numbers and locations are listed on the chain-of-custody (COC) form. Specific details for completing the chain-of-custody form are given in Appendix A-1. When transferring possession of the samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC form. This form documents transfer of sample custody from the sampler to another person, to a mobile laboratory, to the CLP laboratory, to the sample shipper, or to a secure storage area.

4. Shipping containers are secured with strapping tape and EPA custody seals for shipment to the laboratory. The custody seals are covered with clear plastic tape.

5. The samples are sent by common carrier, and a copy of the bill of lading is retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody form.

6. Whenever samples are split with a source or government agency, a separate sample receipt is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted on the form.

7.2 Laboratory Custody Procedures

Laboratory custody procedures are adopted by reference as those required by the Contract Laboratory Program. The purge files from the CLP will be maintained by the Region V CRL laboratory support team data coordinator.

7.3 Evidence File Custody Procedure

The RI files, along with all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews, will be maintained in a secure, limited access area and under custody of the site manager or document custodian.

8.0 CALIBRATION PROCEDURES AND FREQUENCY

All CLP laboratories will be required to conform to the calibration procedures and frequency of calibration schedule required in the Contract Laboratory Program Inorganic Statements of Work, and the Special Analytical Services Request for Fish Tissue Analyses presented as Appendix B.

9.0 ANALYTICAL PROCEDURES

9.1 CLP-SAS

All fish tissue samples collected will be analyzed for manganese and mercury by the CLP. Analytical procedures will conform to guidelines presented in Appendix B, Special Analytical Services Request for Manganese and Mercury in Fish Tissue.

10.0 DATA REDUCTION, VALIDATION AND REPORTING

10.1 CLP-SAS

The test procedures used by SAS will be clearly identified. Bench records and all records of analyses and calculations for samples, blanks, duplicates, spikes, and standards, with resultant instrument inputs or concentration readouts, will be provided by CLP, SAS, along with worksheets used to calculate results. The laboratory analytical data will be reported in accordance with standard CLP protocol as specified in the SAS Regional Request Form (Appendix B). Data validation will be performed by the Laboratory Scientific Support Section (LSSS) and reviewed by ICF personnel. Data validation will be performed in accordance with the standard EPA Region V validation procedures, which are based on the EPA National Functional Guidelines for Evaluating Inorganic Analyses. The raw data collected from project sampling tasks and used in project reports will be identified and will be included in a separate appendix within the final report. Where test data have been reduced, the method of reduction will be described.

Data reporting format will be consistent with the CLP Statement of Work, SOW-7/88 for inorganic analysis except the results will be reported on the basis of wet weight.

10.2 Field Analysis

All field recording sheets, instrument outputs, and worksheets for calculating results will be retained. Summarized raw data will be appropriately identified in reports and included in a separate appendix in the final RI report. Where test data have been reduced, the method of reduction will be described.

11.0 INTERNAL QUALITY CONTROL PROCEDURES

11.1 CLP-SAS

Quality control requirements for each of the CLP, SAS analyses are specified in Appendix B. Field blanks and duplicates will be collected and submitted to CLP, SAS for analysis to determine if any sample contamination is due to field sampling equipment and to check data precision, respectively.

11.2 Field Analyses

Field analyses are performed on site and do not involve samples that are collected and retained. The primary QA/QC objective is to obtain reproducible measurements to a degree of accuracy consistent with limits imposed by analytical methodologies used and the intended use of the data. Quality control procedures will be limited to checking the reproducibility of measurements by taking multiple readings and by calibration of instruments (where appropriate).

11.3 Report Preparation

During report preparation, data reduction and manipulation will occur. All calculations and other forms of data reduction will be checked by the Site Manager, the ICF QA officer, and the CH2M Hill review team leader. In addition, each report will be reviewed by the ICF QA officer and the CH2M Hill review team leader for accuracy and consistency.

12.0 PERFORMANCE AND SYSTEM AUDITS

12.1 CLP-SAS

System audits and required performance limits are specified for the CLP, SAS analysis in Appendix B. Performance and system audits for CLP, SAS, are the responsibility of the Support Services Branch, OERR, EPA and of EMSL - Las Vegas, EPA.

The Quality Assurance Manager (QAM) will monitor and audit performance of the QA procedures to assure that the project is performed in accordance with approved quality assurance procedures. The QAM will conduct the audits as described in Section 8, Quality Control Procedures, of the CH2M Hill REM IV Zone Management Plan. A copy of Section 8, Quality Control Procedures of the CH2M Hill REM IV Zone Management Plan is included in Appendix C. Audits may be scheduled at various times to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field notebooks and sampling procedures.

12.2 Field Analyses

All instruments and equipment used in making field measurements will be regularly calibrated (where appropriate) as specified in Appendix A. Performance/system audits shall be the responsibility of the ICF Field Operations Coordinator and CH2M Hill QA Manager.

13.0 PREVENTIVE MAINTENANCE

CLP preventative maintenance procedures to be utilized include CLP SOW-7/88 for metals analyses.

Preventive maintenance procedures will be carried out on any field equipment utilized during the investigation with overview by the Field Operations Leader and CH2M Hill's Quality Assurance Manager.

14.0 DATA ASSESSMENT AND COMPLETENESS

14.1 CLP-SAS

Data assessment is the responsibility of the Laboratory Scientific Support Section (LSSS) of the CRL. Data completeness will be checked by ICF and the SMO.

Following validation, ICF will assess the data to determine precision, accuracy, and completeness. It is expected that spike samples will give recoveries of 50 - 150%, while duplicates will have RPDs <60%. The limits have been set high due to the type of samples being analyzed and the impact that the variability would have on the subsequent risk assessment to be performed using the data. The actual sampling techniques used by the field crew will be reviewed by the project manager for deviations from the written sampling procedures. If deviations in precision, accuracy, or sampling technique are found, their impact on the data will be assessed.

Lost or suspect data will be evaluated in terms of sample location, analytical data lost (fraction, individual parameter, etc.), decision to be made with the data, and risk associated with an erroneous decision. Critical locations or critical analytical data will be examined to determine their adequacy and impact on the overall objectives of the project.

14.2 Field Analyses

The Quality Assurance Manager (QAM) will assess data to assure QA/QC objectives are met.

15.0 CORRECTIVE ACTION PROCEDURES

15.1 CLP-SAS

If quality control audits result in detection of unacceptable conditions of data, the laboratory will report the problem to the LSSS. The SM, RPM, Ohio EPA Project Coordinator and QAM will participate in determining an acceptable corrective action to the problem. The corrective action shall be mutually agreed upon by USEPA, Ohio EPA, CH2M Hill and ICF. The Field Operations Leader shall be responsible for implementing changes to sampling procedure and for ensuring sampling activities are performed in accordance with established protocol. Corrective actions may include:

- Reanalyzing samples if holding time criteria permit
- Resampling and analyzing
- Evaluating and amending sampling and analytical procedures
- Accepting data acknowledging level of uncertainty

The CLP laboratories are required by USEPA to prepare QA manual and Standard Operating Procedures (SOP) to address these elements and Good Laboratory Practices (GLP) procedures. Performance and system audits of the CLP are the responsibility of the Support Service Branch, OERR, EPA and of EMSL-Las Vegas, EPA. Data assessment will be performed by the Laboratory Scientific Support Section (LSSS) of CRL according to the established assessment procedures as described in "Functional Guidelines for Evaluating Inorganic Analysis," R-582-5-5-01, USEPA, May 28, 1985.

16.0 QUALITY ASSURANCE REPORTS

No separate QA report for this project is anticipated. The final report will contain separate QA sections that summarize data quality information collected during the project. The information to be summarized may include:

- Analytical results of QA/QC samples from the field and analytical laboratories.
- Percent of blank, duplicate and spike determinations.
- Results of intralaboratory precision and accuracy.
- Results of performance and system audits in the field and at the laboratories.
- Data quality assessment.
- Significant quality assurance problems and recommended solutions.

APPENDIX A

SAMPLING PLAN
FISH SPECIES SURVEY AND TISSUE SAMPLING
FIELD INVESTIGATION

REPUBLIC STEEL QUARRY SITE
ELYRIA, OHIO

SAMPLING PLAN

TABLE OF CONTENTS

SAMPLING PLAN
FISH SPECIES SURVEY AND TISSUE SAMPLING
FIELD INVESTIGATION
REPUBLIC STEEL QUARRY SITE
ELYRIA, OHIO

1.0 OBJECTIVE

This sampling plan documents procedures and practices to be used in performing the fish species survey and collecting samples of fish tissue for laboratory analyses from the Republic Steel Quarry Site.

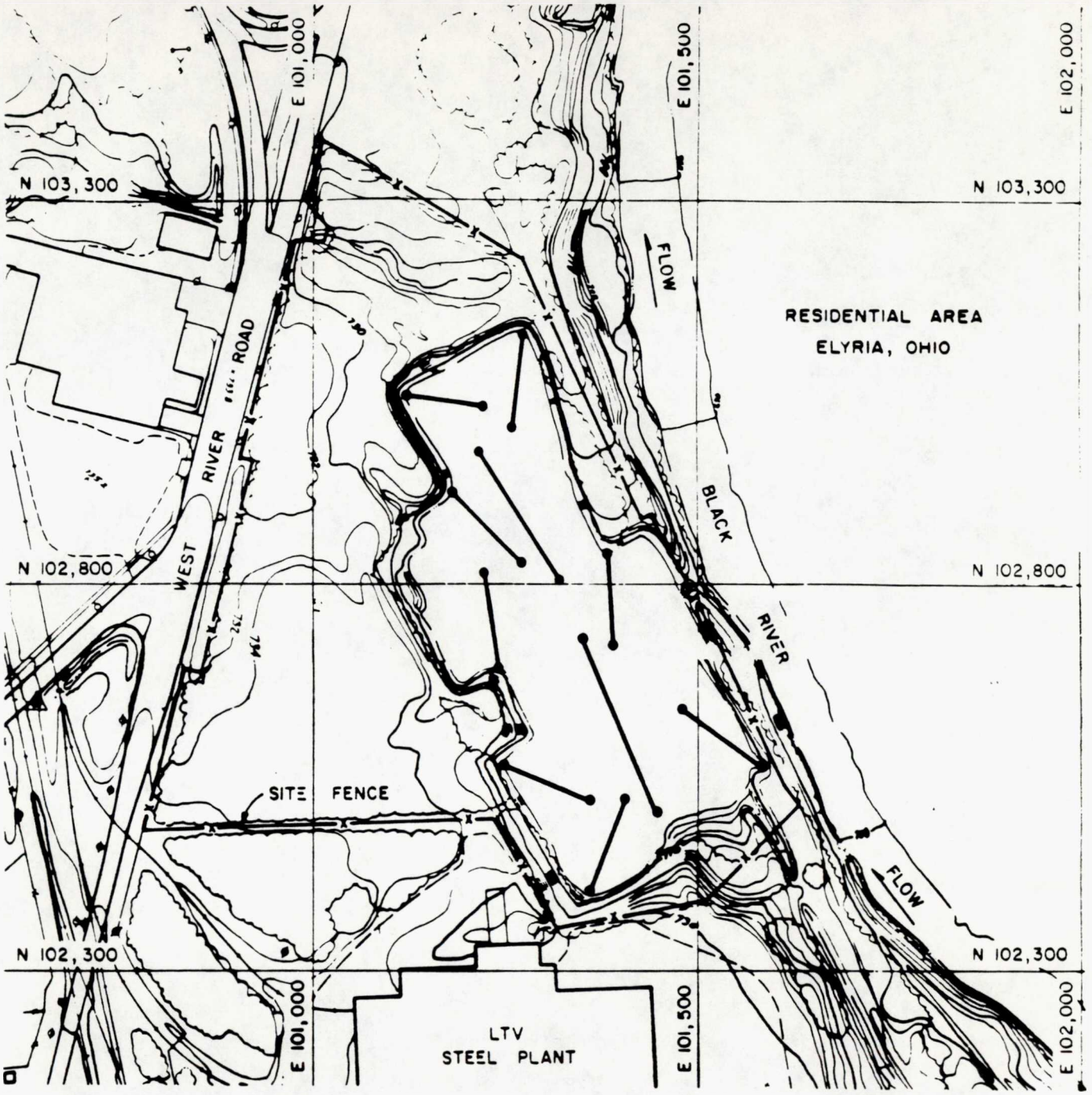
2.0 FISH SPECIES SURVEY AND FISH TISSUE SAMPLING

2.1 Sampling Locations and Depths

Approximately seventeen (17) samples will be collected as a part of this investigation. Nine (9) samples from the quarry, two duplicate samples, two samples from the Black River above the dam, two samples downstream of the site, and two blank samples are anticipated. The proposed sample descriptions and rationale for sample collection are presented on Table A-1. Figure A-1 shows the proposed layout of gill nets to be set in the quarry. Based on bottom contours of the quarry, this layout should enable collection of fish from the surface waters, intermediate zones, and deepest portions of the quarry. For risk comparison purposes, and to evaluate potential impact on Black River fish, four Black River fish samples will also be collected. Two samples will be obtained from above the dam (approximately 6 feet vertical height) to ensure that the fish have not previously been in contact with quarry water discharges and then swam upstream. Two other fish samples will be collected downstream of the quarry discharge point in pooled areas of the Black River.

2.2 Sampling Method

Fish will be collected from the quarry and Black River using gill nets and/or electroshocking methods. The gill nets consist of panels usually ranging in screen width from 1/2 inch to 3 inches. The gill nets will be placed as shown on Figure A-1, and will capture fish from 0 to 6 feet above the bottom of the quarry where they were set. Gill net locations were selected to capture fish from the deepest portions of the quarry where some bottom feeders may exist, intermediate zones, and shallow areas where surface fish exist. Gill nets will be set and allowed to capture fish overnight prior to pulling up and emptying the nets. The exact number and locations of nets placed within the quarry may be modified by the Field Operations Leader as necessary to ensure that a reasonable quantity of fish are captured. Possibly the nets will be set for more than one night, if additional biomass is required. If necessary, electroshocking will be attempted to collect fish from surface waters of the quarry.



SOURCE: CITY OF ELYRIA
ENGINEERING DEPARTMENT
SCALE: 1" = 100'

LEGEND:

125 FT. LENGTH GILL NET

250 FT. LENGTH GILL NET

N

0 200 400

SCALE IN FEET

FIGURE A-1
FISH SAMPLING/GILL NET LOCATIONS
REPUBLIC STEEL QUARRY SITE

TABLE A-1
PROPOSED SAMPLE DESCRIPTIONS AND RATIONALE

SAMPLE NO.	PROPOSED SAMPLE DESCRIPTION	SAMPLING RATIONALE
RSQ-FT-001	Bass Species 1, Small, Fillet	Endangerment Assessment
RSQ-FT-002	Bass Species 1, Large, Fillet	Endangerment Assessment
RSQ-FT-003	Bass Species 2, Small, Fillet	Endangerment Assessment
RSQ-FT-004	Bass Species 2, Large, Fillet	Endangerment Assessment
RSQ-FT-005	Catfish, Small, Fillet	Endangerment Assessment
RSQ-FT-006	Catfish, Large, Fillet	Endangerment Assessment
RSQ-FT-007	Carp, Small, Fillet	Endangerment Assessment
RSQ-FT-008	Carp, Large, Fillet	Endangerment Assessment
RSQ-FT-009	Sunfish, Large, Fillet	Endangerment Assessment
RSQ-FT-010	Bass, Large, Fillet (Duplicate)	Quality Assurance Sample
RSQ-FT-011	Bass, Large, Fillet (Duplicate)	Quality Assurance Sample
RSQ-FT-012	"Contaminant Free" Fish, Fillet, (Blank)	Quality Assurance Sample
RSQ-FT-013	"Contaminant Free" Fish, Fillet, (Blank)	Quality Assurance Sample
RSQ-FT-014	Black River Above Dam, Bass, Large, Fillet	Risk Comparison Sample
RSQ-FT-015	Black River Above Dam, Carp, Large, Fillet	Risk Comparison Sample
RSQ-FT-016	Black River Below Site, Bass, Large, Fillet	Risk Comparison Sample
RSQ-FT-017	Black River Below Site, Carp, Large, Fillet	Risk Comparison Sample

The fish in the Black River may be sampled using a multigear approach utilizing gill nets, electroshocking, seines and traps. Samples will be taken above and below the dam.

2.3 Fish Species Identification

All fish collected will be identified to the species level by trained personnel familiar with fish identification. The fish will be cross-referenced against published materials, including Trautman's Fishes of Ohio, to ensure correct identification has been made. During fish processing, fish will also be weighed and lengths measured. The fish will be set in groups by species on a clean aluminum foil surface (dull side up) following species identification and size measurements. This process will aid in selection of fish for laboratory analyses (see Section 2.4). One whole fish of each species identified will be retained and stored in a 10% formaldehyde solution for at least a two-year period to allow fish species confirmation if it becomes necessary at a later date during the project.

2.4 Fish Selection for Tissue Analyses

Following identification of the fish species and measurement of fish lengths and weights, a subset of fish will be selected that will meet the following criteria:

- The fish will be of the same species, and approximately the same length and weight.

- The subset will be representative of the majority of the fish of that species and size (i.e., not only the unhealthy appearing or healthy appearing fish will be chosen for analyses).

- At least five fish will be selected, filleted, and homogenized to ensure that a representative composite sample is obtained.

- More than five fish may be chosen as necessary to ensure that a sufficient volume of sample is obtained to fill sample jars for performing inorganic analyses.

Once the fish subset has been selected, the fish will be wrapped in aluminum foil (with the dull side toward the fish), and the fish package will be marked with the sample identification number (see Section 4.0) using permanent markers to write on duct tape to be placed on the foil package.

The fish packages will be placed on dry ice to freeze, the packages will be placed in coolers containing dry ice, the coolers will be closed and custody sealed, and the fish will be returned to Aquatic Systems Corporation's laboratory for homogenization. Upon arrival at Aquatic Systems Corporation, the samples will be removed from the coolers and placed in large freezers. The freezers will be set at a temperature below zero degrees Celcius. The

freezers will be closed and custody seals applied, and the laboratory security system will be activated overnight, until the samples can be prepared as described in the following section.

2.5 Fish Tissue Sample Preparation/Homogenization Method

Each fish will be filleted (with skin remaining) and the scales removed. The fish will then be placed in a decontaminated grinder or blender and homogenized. The homogenized fish sample will be placed into two 8 ounce soil jars for laboratory analyses, allowing 10% at the top of the jars for expansion. The samples will then be documented for CLP analyses in accordance with Section 6.0, and placed in a freezer until shipment to the lab. The matrix spike and matrix spike duplicate samples will be prepared at the laboratory using the procedure detailed in the SAS request. To ensure blank samples are not used for matrix spike/matrix spike duplicate analyses, the samples to be used for matrix spike/matrix spike duplicate analyses will be specified on the sample tags, Special Analytical Services Packing List, and Chain of Custody form. Extra sample volume will be provided for samples designated for matrix spike/matrix spike duplicate analyses.

A sufficient number of fish shall be collected to ensure that at least two 8 ounce jars of homogenized fish fillet sample may be obtained. One additional 8 ounce jar will be provided for each matrix spike and matrix spike duplicate sample. In the event that too few fish of a given size and species are collected to fill two 8 ounce jars with filleted material, the specific sample may be eliminated or replaced with another size and species of fish. This will be sufficient for purposes of this investigation; if so few fish exist that two 8 ounce jars cannot be filled, then the exposure scenario from consumption of this fish type is highly unrealistic. Too few fish would exist to support the high consumption, long term exposure scenario used in calculating fish ingestion risks.

3.0 DECONTAMINATION

3.1 Sample Collection Equipment

Sample collection equipment, such as gill nets and electroshocking probes, will be thoroughly cleaned by scrubbing with a trisodium phosphate (TSP) or alconox decontamination fluid followed by a potable water rinse. The TSP or alconox decontamination fluid will be potable water with approximately 2.5 percent (TSP or alconox) dissolved (by weight).

3.2 Sample Preparation/Homogenization Equipment

Sample preparation/homogenization equipment, such as grinders, blenders and mixing bowls, will be carefully decontaminated before each sample by scrubbing with a TSP or alconox decontamination fluid, followed by a potable water rinse

as described above for sample collection equipment. Then, the equipment will be washed in a solution of 10% nitric acid in distilled water, and thoroughly rinsed with distilled water.

4.0 SAMPLE NUMBERING SYSTEM

A sample numbering system will be used to identify each sample for chemical analysis, including duplicates and blanks. A listing of the sample identification numbers will be maintained in the log book by the Sample Team Leader. Each sample number will consist of three components described below:

4.1 Project Identification

A three-letter designation will be used to identify the site where the samples were collected. For this project, it will be RSQ for Republic Steel Quarry.

4.2 Sample Location

Each sample collected will be further identified by an alpha-code corresponding to the sample type. The alpha-code is as follows:

FT - Fish Tissue

Field blanks and duplicate samples will not have any special identification code so that laboratories will be unable to distinguish them from other samples for analysis. To ensure blank samples are not used for matrix spike/matrix spike duplicate analyses, the samples to be used for matrix spike/matrix spike duplicate analyses will be specified on the sample tags, Special Analytical Services packing list, and Chain of Custody forms. Extra sample volume will be provided for samples designated for matrix spike and matrix spike duplicate analyses. One extra 8 ounce jar will be provided for each matrix spike and matrix spike duplicate sample.

4.3 Sample Identifier

A three-digit number will be used to indicate sample collection location.

4.4 Sample Number Examples

- RSQ-FT-001 - Republic Steel Quarry Fish Tissue Sample Number 1
- RSQ-FT-002 - Republic Steel Quarry Fish Tissue Sample Number 2

Duplicates and field blanks will be prepared and numbered uniquely as if they were separate samples. A record of identification numbers for samples, blanks and duplicates, with sample location, date and time of collection, will be maintained by the sample team leader.

5.0 SAMPLE ANALYSES TO BE PERFORMED

Below is a listing of analyses to be conducted:

- Fish Tissue Samples
  - U. S. EPA CLP:Special Analytic Services Manganese and Mercury in Fish Tissue

All samples will be considered low-concentration samples. The determination of low concentration is based on existing analytical data collected from the site.

6.0 SAMPLE DOCUMENTATION

All samples will be collected under chain-of-custody procedures. Standard paperwork, including sample tags, SAS packing lists, chain-of-custody forms, and custody seals used for CLP sample tracking and records will be filled out. All pertinent information about the samples will be logged in the site log maintained by the Team Leader. This information will include sample time, location, tag numbers, designation, and sampler. New readings, weather conditions, and field modifications or decisions will also be recorded. The log book will be filled in ink unless weather conditions dictate otherwise. Photographs with the time, date, location, and task description will also be noted in the log book. The documentation procedures are outlined in Appendix A-1.

Following documentation, the samples are replaced in the deep freeze chest and custody sealed. When the samples are to be shipped to the laboratory, the samples are removed from the freezer and wrapped in aluminum foil, packed into coolers and shipped via Federal Express overnight delivery. Sample packaging and shipment procedures are also outlined in Appendix A-1.

7.0 WASTE DISPOSAL

All protective clothing and sampling-related wastes will be disposed of in double-bagged plastic garbage bags and placed in an onsite dumpster for subsequent disposal at a municipal landfill. The fish wastes and protective clothing are not anticipated to contain sufficient contaminant concentrations to warrant more stringent disposal measures.

APPENDIX A-1
SAMPLE DOCUMENTATION

1.0 INSTRUCTIONS FOR FILLING OUT DOCUMENTATION

All samples collected at Superfund sites for laboratory analysis must follow established documentation protocol. Adherence to this protocol provides a network of valuable information documenting sample identification and tracking as well as chain-of-custody.

1.1 GENERAL DOCUMENTATION PROCEDURES

Organization and concentration are the keys to completing the required documents efficiently and without error. Make certain that a suitable work area has been set aside with ample table and floor space available for the processing of forms and the packaging of samples. This is especially important for large projects.

Forms, stages, etc. can be filled out in any order; however, past experience has shown that this paperwork can be completed most efficiently and accurately if the sample identification matrix is completed before or in conjunction with the completion of the rest of the documentation.

Sections 1.2 through 1.10 discuss the proper completion of each document. Use these pages as a reference while following this suggested plan of attack:

1. Make or obtain a list of the samples to be packaged and shipped that same day and the laboratories to be used.

2. Enter the case number, CRL number, matrix, sample numbers, laboratory, date sampled and date shipped for each sample on the matrix. NOTE: If portions of a given sample are to be shipped to different laboratories (for organic and inorganic analysis for instance), two entry lines will be required for that sample number to accommodate the chain-of-custody record, airbill, and traffic report numbers corresponding to each portion of the sample.

3. Obtain the QC lot numbers of the prelabeled containers for each sample and enter these on the matrix.

4. Determine the number of shipping containers (coolers) required to accommodate the day's shipment. This is based on the number of samples to be shipped, the number of containers per sample, the number of sample containers that will fit in each cooler, and the number of laboratories to be used. (Note: A group of containers for a single sample should not be split between coolers except when one portion of the sample is to be sent to one laboratory for one type of analysis and the other portion is to be sent to another laboratory for another type of analysis.)

5. Complete an airbill for each laboratory address. (Note: Several coolers may be shipped to the same address under one airbill.) Shipment of medium and high concentration samples requires the use of a special airbill, including a shipper's certification for restricted articles.

6. Enter the airbill numbers on the matrix.

7. Mentally assign a chain-of-custody record to each cooler and determine which sample containers will be shipped in each. (Note: More than one chain-of- custody record may be needed to accommodate the number of samples to be shipped in one cooler.)

8. Assign chain-of-custody numbers to each sample by entering these numbers on the matrix. (Reminder: Portions of samples for organic and inorganic analysis will usually be sent to separate laboratories. Use one line on the matrix for the organics portion information and another line for the inorganics portion information.)

9. Assign tag numbers to each sample container for each sample and enter these numbers on the matrix.

10. Complete SAS packing lists or CRL basic data sheets based on the information provided on the matrix.

11. Complete sample tags based on the information provided on the matrix and the parameters of analysis. Place tags in groups by sample number.

12. Complete chain-of-custody records based on the information provided on the matrix.

13. Assign two custody seals to each cooler. Enter the serial numbers of the seals in the "REMARKS" section of each chain-of-custody form and temporarily clip seals to the form.

14. Group all the paper work associated with each cooler in a separate clip.

15. Obtain full signatures of the STL and initials of significant field team members (including yourself) on the sample tags and at the top of the chain- of-custody forms.

16. Prepare to package samples for shipment.

Following are step-by-step instructions for completing each form. The sample identification code to be used is the sample number as described in Appendix A, Section 4. Other items should be self-evident from the instructions.

1.2 SAMPLE IDENTIFICATION MATRIX (Figure A-2):

1. Indicate site name.
2. Indicate project number.
3. Enter the case number.
4. Enter the CRL number.
5. Specify the sample matrix using the two digit codes listed below followed by the letter (L) to indicate low concentration:
   - FT - Fish Tissue
6. Indicate the sample number.
7. Indicate the chain-of-custody report number.
8. Indicate the laboratory to be doing the analysis.
9. Enter the date the sample was taken - month, day, year (no hyphen or slash, e.g., 051289).
10. Enter the shipping date.
11. Enter the airbill number of the shipment.
12. List sample tag numbers.
13. List the QC lot numbers of the containers.

Note: Data recorded on this form must be suitable for computer entry. Each entry must be left justified and must not exceed the number of digits allotted in each section. If portions of samples are to be sent to more than one laboratory for analysis, allow an entire line for each laboratory to accommodate for the additional chain-of-custody and airbill numbers.

CH2M HILL
REM/FIT
SAMPLE IDENTIFICATION MATRIX

CASE NUMBER	CRL NUMBER	MATRIX	SAMPLE NUMBER	OTR OR MHTR	ITR	CHAIN OF CUSTODY	LAB	DATE SAMPLED	DATE SHIPPED	AIRBILL NUMBER	SAMPLE TAG NUMBERS	QC LOT NUMBERS

JOB NUMBER ____________

SITE NAME ____________

FIGURE A-2

1.3 SAMPLE TAG (Figure A-3)

1. Enter the first six digits of the CRL sample identification.
2. Enter the last three digits of the CRL identification code.
3. Enter date of sampling.
4. Enter time of sampling (military time only).
5. Specify "grab" or "composite" sample with an "X".
6. Insert sample identification code.
7. Obtain signature of sample team leader.
8. Indicate presence of preservative with an "X".
9. Specify parameters for analysis with an "X".

10a. Indicate SAS number.

10b. Indicate case number.

11. Leave BLANK (for laboratory use only).
12. Enter any desired analyses not listed on menu provided (e.g., PCB's, ammonia, sulfide, etc.) and mark box with an "X".

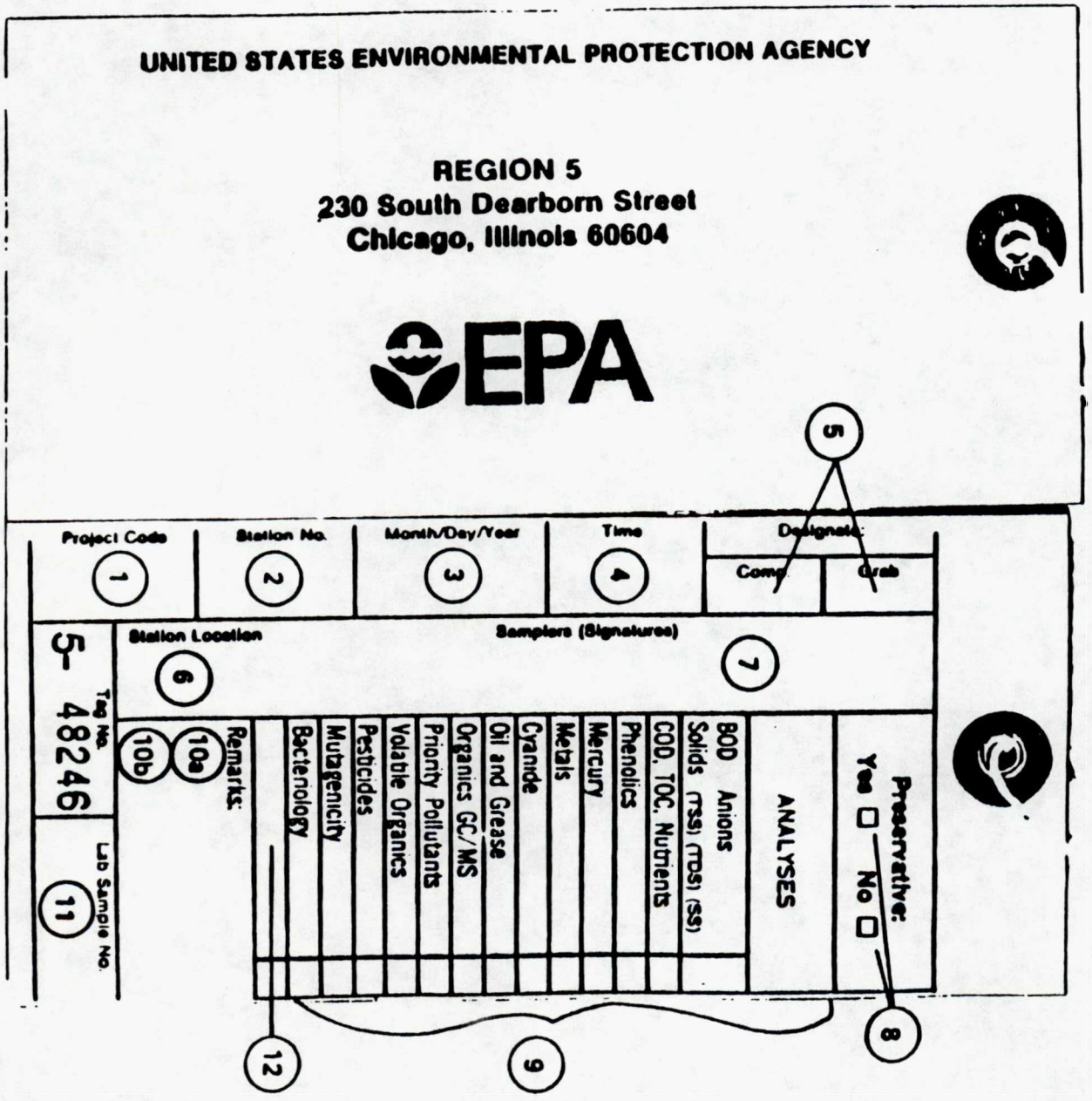

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 South Dearborn Street
Chicago, Illinois 60604
EPA
5
Project Code
1
Station No.
2
Month/Day/Year
3
Time
4
Designate:
Comp
Grab
Station Location
6
Samplers (Signatures)
7
5-
Tag No.
48246
LAB Sample No.
11
Remarks:
10a
10b
Preservative:
Yes
No
8
ANALYSES
BOD Anions
Solids (TSS) (TDS) (SS)
COD, TOC, Nutrients
Phenolics
Mercury
Metals
Cyanide
Oil and Grease
Organics GC/MS
Priority Pollutants
Volatile Organics
Pesticides
Mutagenicity
Bacteriology
9
12

FIGURE A-3

1.4 SAS PACKING LIST (Figure A-4)

1. Insert assigned SAS case number.
2. Insert EPA region number (e.g., V).
3. Insert sample team leader's name.
4. Insert sample team leader's office telephone number (do not use field office telephone number).
5. Insert date sample was taken.
6. Indicate date of shipment.
7. Insert site name.
8. Insert laboratory name and address.
9. Indicate name of laboratory contact.
10. List SAS sample numbers, which should include the SAS number.
11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for PCB analysis, tag number 5-48246).
12. Leave BLANK - for laboratory use only.

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

(1) SAS Number

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office: (2)	Sampling Date(s): (5)	Ship To: (8)	For Lab Use Only
Sampling Contact: (3) (name)	Date Shipped: (6)		Date Samples Rec'd:
(4) (phone)	Site Name/Code: (7)	Attn: (9)	Received By:

	Sample Numbers (10)	Sample Description i.e., Analysis, Matrix, Concentration (11)	Sample Condition on Receipt at Lab (12)
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			
17.			
18.			
19.			
20.			
			For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

FIGURE A-4

1.5 CHAIN-OF-CUSTODY FORM (Figure A-5)

1. Enter first six digits of the CRL sample identification code.
2. Enter site name and CH2M Hill project number.
3. Obtain full signature of sample team leader and signed initials of active team members (including paperwork person).
4. Enter last three digits of the CRL sample identification code.
5. List sampling dates for all samples.
6. List sampling times for all samples.
7. Indicate "grab" or "composite" sample with an "X."
8. List sample numbers.
9. Enter number of containers per sample and container volume (e.g., 2-40 ml).
10. List analyses individually.
11. Construct column heading for SAS number.
12. Construct column heading for "tag number" and list tag numbers for each sample container.
13. Obtain signature of sample team leader and carry out chain of custody procedures.
14. State carrier service and air bill number, lab service, and custody seal numbers.
15. Write in the words "CASE #:" and enter the case number.

CHAIN OF CUSTODY RECORD

PROJ. NO. (1)	PROJECT NAME (2)	NO. OF CONTAINERS (10)	REMARKS (15)
SAMPLERS: *(Signature)* (3)			

STA. NO. (4)	DATE (5)	TIME (6)	COMP (7)	GRAB (7)	STATION LOCATION (8)	NO. OF CONTAINERS (9)	REMARKS (11, 12)

Relinquished by: *(Signature)* (13)	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks (14)	

Distribution: White – Accompanies Shipment; Pink – Coordinator Field Files; Yellow – Laboratory File

1.6 NOTICE OF TRANSMITTAL (Figure A-6)

1. Enter name of team leader.
2. Enter "ICF Technology Inc."
3. Enter case number.
4. Complete date.
5. Enter number of samples shipped.
6. Enter matrix of samples.
7. Enter "Republic Steel Quarry."
8. Enter "Elyria, Ohio."
9. Enter date.
10. Enter name and address of CH2M Hill Sample Documentation Coordinator.

NOTICE OF TRANSMITTAL

DATE:

TO:

FROM: (1) Name / (2) Firm

CH2M HILL PROJECT NO.:

Enclosed are appropriate copies of the sample documentation forms completed under Case # (3) for the (4), 19(4), shipment of (5) Quantity (6) Matrix samples from the (7) site located in (8), (8).

FIGURE A-6

NOTICE OF TRANSMITTAL

1.7 CENTRAL REGIONAL LABORATORY (CRL) SAMPLE DATA REPORT FORM (Figure A-7)

The Central Regional Laboratory Sample Data Report Form (Figure A-7) is not filled out by field personnel. This form is completed by CH2M Hill's Sample Documentation Coordinator from copies of the Chain-of-Custody forms which are submitted to the Sample Documentation Coordinator. This CRL Sample Data Report is similar to the Sample Identification Matrix except that it is specifically for CRL samples.

1.8 RECEIPT FOR SAMPLES FORM

A completed Receipt for Samples Form will be used whenever splits are provided to other parties. This form must be completed and a copy given to the other party. The original will be retained in the project files. At potential source sites, splits of all samples collected must be offered to an official at the site. If the splits are declined, the Receipt for Samples Form should be so marked.

1.9 FIELD LOG BOOK

All information pertinent to a field survey or sampling effort will be recorded in a log book or equivalent standardized form. Each page/form will be consecutively numbered and will be at least 4-1/2 in. by 7 in. in size. All entries will be made in indelible ink or hard lead pencil and all corrections will consist of line-out deletions that are initialed and dated. As a minimum, entries in a log book will include the following:

- Purpose of sampling.
- Location, description, and log of photographs of the sampling point.
- Details of the sampling site (for example, the elevation of the casing, casing diameter and depth, integrity of the casing, etc.).
- Name and address of field contact.
- Documentation of procedures for preparation of reagents or supplies which become an integral part of the sample (e.g., filters and absorbing reagents).
- Identification of sampling crew members.
- Type of sample (for example, groundwater, soil, sludge, or waste water).
- Suspected waste composition.
- Number and volume of sample taken.
- Sampling methodology, including distinction between grab and composite samples.
- Sample preservation.
- Date and time of collection.
- Collector's sample identification number(s).
- Sample distribution and how transported (for example, name of the laboratory and cartage agent-Federal Express, United Parcel Service
- References such as maps of the sampling site.

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT
ORGANICS/INORGANICS
THIS FORM IS TO BE USED FOR SAMPLES SENT TO CONTRACT ONLY

CASE NUMBER/SAS No _____ SITE NAME _____ LABORATORY _____ DATE SHIPPED _____

SUPERFUND DU NUMBER _____ EPA RPM or OSC (S M S)/(CES) _____ CERCLIS NUMBER _____ PAGE _____ OF _____

ACTIVITY NUMBER

CRL LOG NUMBER	ORGANIC TRAFFIC REPORT NUMBER or SAS Packing List No.	INORGANIC TRAFFIC REPORT NUMBER	WATER OR LIQUIDS												SEDIMENTS or SOILS											
			ACID/BASE NEUTRAL CPDS ORGANIC SCAN UG L TOX17874	VOLATILE ORGANIC ANALYSIS ORGANIC SCAN UG L TOX17864	WATER POLYCHLORINATED BIPHENYLS UG L PES 17144	WATER CHLORINATED PESTICIDES UG L PES17134	TOTAL METALS IN WATER UG L MET1111	WATER CYANIDE UG L MIN74819	NITRATE NITRITE MG L MIN7284	AMMONIA MG L MIN7284	RESIDUE FILTERABLE TDS MG L MIN7382	RESIDUE NON FILT TSS MG L MIN7372			ACID BASE NEUTRAL CPDS ORGANIC SCAN MG KG TOX215722	VOLATILE ORGANIC ANALYSIS ORGANIC SCAN MG KG TOX215822	SEDIMENTS POLYCHLORINATED BIPHENYLS MG KG PES211422	SEDIMENT CHLORINATED PESTICIDES MG KG 211322	TOTAL METALS MG KG MET413	CYANIDE MG KG MIN44830	EP TOXICITY METALS MG KG	AMMONIA MG KG MIN42825				

FIGURE A-7

- Any field measurements made (for example, pH, specific conductance, temperature, and water depth).
- Signature and date by the personnel responsible for observations.
- Decontamination procedures.

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a log book or standardized form. However, records will contain sufficient information so that someone can reconstruct the sampling activity without relying on the collector's memory. The log book and standardized forms will be kept under strict chain-of-custody.

1.10 CORRECTIONS TO DOCUMENTATION

Unless prohibited by weather conditions, all original data recorded on Traffic Report Forms, Sample Identification Tags, Chain-of-Custody Records, and Receipt for Sample Forms will be written with waterproof ink. No accountable serialized documents are to be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document.

If an error is made on an accountable document assigned to one individual, that individual shall make corrections by making a line through the error and entering the correct information. The erroneous information should not be obliterated. Any subsequent error discovered on an accountable document should be corrected by the person who made the entry. All subsequent corrections must be initialed and dated.

1.11 LABORATORY CUSTODY

Laboratory custody will conform to procedures established for the CLP. These procedures include:

- Designation of a sample custodian.
- Correct completion by the custodian of the chain-of-custody record, sample tag, and laboratory request sheet (including documentation of sample condition upon receipt).
- Laboratory sample tracking and documentation procedures.
- Secure sample storage (of the appropriate environment - refrigerated, dry, etc.).
- Proper data logging and documentation procedures including custody of all original laboratory records.

2.0 PACKING AND SHIPPING PROCEDURES

Sample packaging and shipping procedures are based on U.S. EPA Specifications, as well as Department of Transportation (DOT) regulations (40 CFR). The procedures vary according to sample concentration and matrix and are designed to provide optimum protection of samples and the public.

All samples will be shipped within 48 hrs. of collection or before 50027 of the holding time has elapsed. Shipping containers must be insulated, durable, and watertight. Bagged samples (to prevent vermiculite contamination of samples, all containers regardless of size/type must be placed inside a sealed plastic bag before packing in vermiculite/zonolite) are to be cushioned within the shipping container with vermiculite packing material (Zonolite). Preformed poly-foam cooler liners are available for shipment of low-concentration samples only.

Following shipment, airbill numbers must be called in to the SMO and to the sample documentation coordination.

Step-by-step packing instructions are provided below.

2.1 LOW-CONCENTRATION SAMPLES

1. Prepare cooler(s) for shipment.
   - Tape drain(s) shut.
   - Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
   - Place mailing label with laboratory address on top of cooler(s).
   - Fill bottom of cooler(s) with about 3 in. of vermiculite or use preformed poly-foam liner (low concentration only).
   - Place appropriate traffic reports, SAS packing lists, or Regional field sheets and chain-of-custody records with corresponding custody seals on top of each cooler.
2. Arrange decontaminated sample containers in groups by sample number.
3. Mark volume levels on bottles with a grease pencil.
4. Secure appropriate sample tags around caps/lids of containers with string or wire.
5. Secure container caps/lids with strapping tape.
6. Arrange containers in front of assigned coolers.
7. Affix appropriate adhesive labels from assigned traffic report to each container. Protect with clear label protection tape.
8. Seal each container within a separate plastic bag.
9. Arrange containers in coolers so that they do not touch.
10. If ice is required to preserve the samples, cubes should be repackaged in double zip-loc bags and placed on and around the containers (especially on VOA vials).
11. Fill remaining space with vermiculite (or place poly-foam liner cover on top of samples).
12. Sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express, Purolator, or Emery.
13. Separate copies of forms. Seal proper copies within a large zip-loc bag and tape to inside lid of cooler. Distribute remaining copies as indicated in Section 2.2.

14. Close lid and latch.
15. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.
16. Tape cooler shut on both ends, making several complete revolutions with strapping tape (do not cover custody seals). See Figure A-8 for an illustration of a cooler ready for shipment.
17. Relinquish to Federal Express. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator, along with the other documentation.
18. Telephone the Sample Management Office in Alexandria, Virginia. (NOTE: this step should be omitted for samples sent to the CRL).
    Mr. Tony Nesky
    (703) 557-2490
    Provide the following information:
    - Your name
    - Project name
    - Case number
    - Number of samples sent to each laboratory for analysis
    - Airbill numbers

    This must be done immediately following sample shipment. (If the SMO is closed at that time, call in the information first thing the next day.)

2.2 DISTRIBUTION OF COMPLETED DOCUMENTS

Final disposition of the completed documents is as follows:

- Shipped with Samples:
  Chain-of-custody form, white original
  Traffic report forms, white and yellow copies
  SAS packing list, pink and gold copies
  Sample tags
- Retained by RI Project Manager:
  Sample identification matrix
  Field log books (at completion of project)
- Sent to CH2M Hill Documentation Coordinator:
  Chain-of-custody form, pink and yellow copies
  Traffic report forms, white original and pink copy
  SAS packing list, white original and yellow copy
  Notice of transmittal

2.3 SPECIAL INSTRUCTIONS FOR SHIPPING SAMPLES VIA FEDERAL EXPRESS

1. Label cooler as hazardous shipment.
   - Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
   - Write or affix sticker saying "This Side Up" on two adjacent sides.

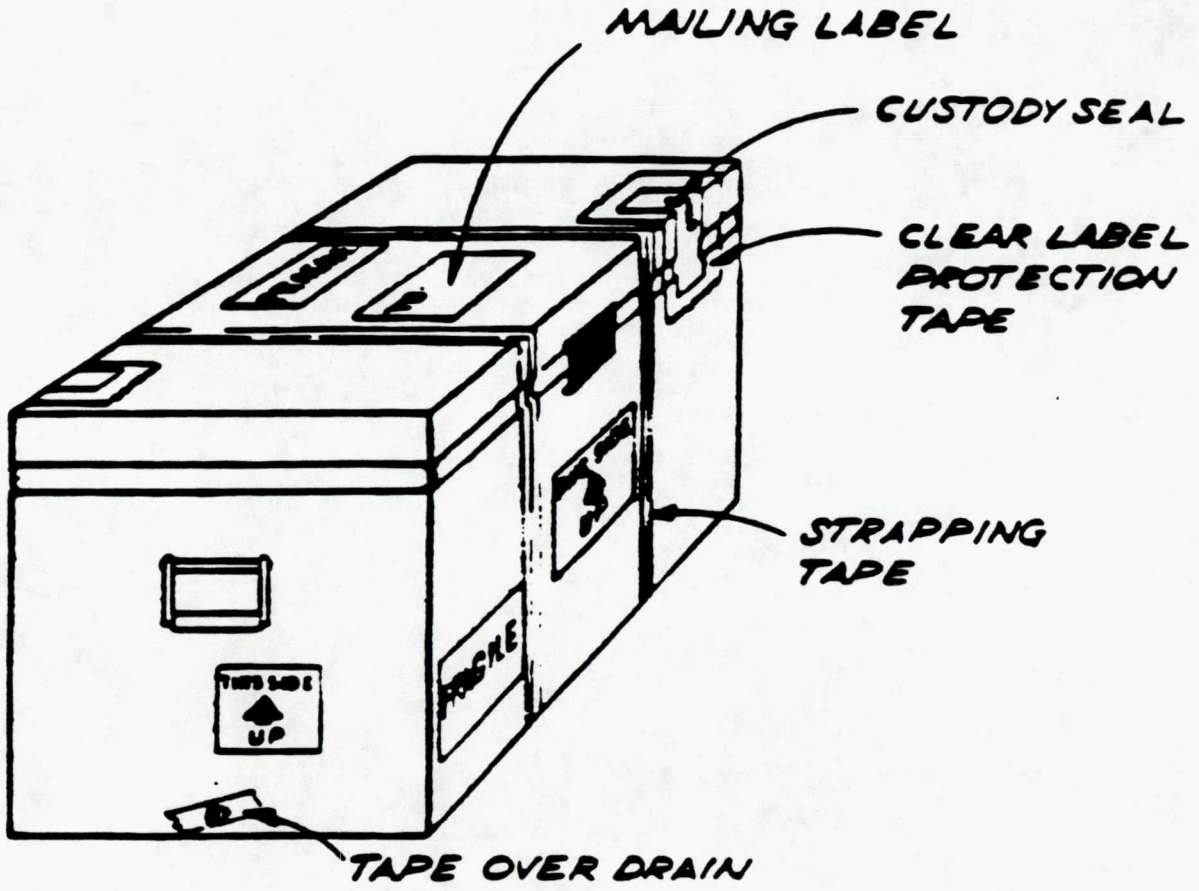

MAILING LABEL
CUSTODY SEAL
CLEAR LABEL PROTECTION TAPE
STRAPPING TAPE
THIS SIDE UP
FRAGILE
TAPE OVER DRAIN

FIGURE A-8

- Write or affix sticker saying "ORM-E" with box around it on two adjacent sides. Below ORM-E, write NA#9188.
- Label cooler with "Hazardous Substance, NOS.", and "liquid" or "solid", as applicable.

2. Complete the special shipping bill for restricted articles (Figure B-15 A-9).
   - Under Proper Shipping Name, write "Hazardous Substance, NOS." and "liquid" or "solid", as applicable.
   - Under Class, write "ORM-E."
   - Under Identification No. write NA#9188.

3. If samples are believed to be environmental samples having low contamination, the samples may be shipped using standard Federal Express airbills, and the coolers will not require special marking.

FEDERAL EXPRESS

USE THIS AIRBILL FOR DOMESTIC SHIPMENTS WITHIN THE CONTINENTAL U.S.A., ALASKA AND HAWAII.
DO NOT USE FOR PUERTO RICO OR INTERNATIONAL DESTINATIONS.
COMPLETE PURPLE AREAS. SEE BACK OF AIRBILL FOR MORE INSTRUCTIONS.
QUESTIONS? CALL 800-238-5355 TOLL FREE.

AIRBILL NUMBER 7099267125

75 80c1M 7099267125

7 0 9 9 2 6 7 1 2 5

PRESS HAI COPIES PLEASE PRINT OR TYPE

ORIGIN COPY

Sender's Federal Express Account Number | Date

1 From (Your Name) (Please Print) | (Your Phone Number) (very important)

Company | Department/Floor No

Street Address

City | State | ZIP (Required For Correct Invoicing)

2 To (Recipient's Name) (Please Print) | (Recipient's Phone Number) (very important)

Company | Department/Floor No

Exact Street Address (Use of P.O. Boxes or P.O. # Zip Codes Will Delay Delivery And Result in Extra Charges.)

City | State | ZIP (Street Address Zip Required)

3 YOUR BILLING REFERENCE INFORMATION (FIRST 24 CHARACTERS WILL APPEAR ON INVOICE.)

PAYMENT ☐ Bill Sender ☐ Bill Recipient's FedEx Acct. No. (Fill in Account Number below) ☐ Bill 3rd Party FedEx Acct. No. (Fill in Account Number below) ☐ Bill Credit Card (Fill in Credit Card Number below) ☐ Cash

4 SERVICES (CHECK ONLY ONE BOX)

1 ☐ PRIORITY 1 Overnight Delivery Using Your Packaging. When using ICAO regulations, please mark appropriate box(es). Do not mark for CFR 49.
☐ Cargo Aircraft only
☐ Dangerous goods as per attached Dangerous Goods Transport Document

5 ☐ STANDARD AIR Delivery not later than second business day

SERVICE COMMITMENT

PRIORITY 1: Delivery is scheduled early next business morning in most locations. It may take two or more business days if the destination is outside our primary service areas.
STANDARD AIR: Delivery is generally next business day or not later than second business day. It may take three or more business days if the destination is outside our primary service areas.

DELIVERY AND SPECIAL HANDLING (CHECK SERVICES REQUIRED)

1 ☐ HOLD FOR PICK-UP (Fill in Section H at right)
2 ☐ DELIVER WEEKDAY
3 ☐ DELIVER SATURDAY (Extra charge)
4 ☒ DANGEROUS GOODS (1st and Standard Air Packages only. Extra charge)
5 ☐ CONSTANT SURVEILLANCE SERVICE (CSS) (Extra charge. Do not Complete Section 5)
6 ☐ DRY ICE _____ Lbs.
7 ☐ OTHER SPECIAL SERVICE _____
8 ☐
9 ☐ SATURDAY PICK-UP (Extra charge)
10 ☐

PACKAGES	WEIGHT	YOUR DECLARED VALUE (See right)	OVER SIZE
Total	Total	Total	

Received At
1 ☐ Regular Stop
2 ☐ On-Call Stop
3 ☐ Drop Box 4 ☐ BSC 5 ☐ Station

Federal Express Corp. Employee No.

Date/Time For Federal Express Use

H HOLD FOR PICK-UP AT THIS FEDERAL EXPRESS LOCATION: Street Address (See Service Guide or Call 800-238-5355)

City | State

ZIP (Zip Code of Street Address Required)

YOUR DECLARED VALUE

DAMAGE OR LOSS

We will pay for no more than $100 per package in the event of physical loss or damage unless you claim a higher Declared Value in the left and document higher actual loss in the event of a claim. We charge 30¢ for each additional $ 100 declared value up to the maximum of $ 500. See our Service Guide. Declared value restrictions are shown on the back of the Sender's Copy of this airbill. We make no expressed or implied warranties.

DELAY

There is always a risk of late delivery or non-delivery. In the event of late delivery, Federal Express will, at your request and with some limitations, refund air transportation charges paid. See back of Sender's Copy of this airbill for further information.

CONSEQUENTIAL DAMAGES

We will not be responsible or liable for any loss or damage resulting from delay, non-delivery or damage to a package except as noted above. This includes loss of sales, income, interest, profits, attorney fees and other costs but is not limited to these items. Such damages are called consequential damages.

DO NOT SHIP CASH OR CURRENCY

Federal Express Use	
Base Charges	
Declared Value Charge	
Origin Agent Charge	
Other	
Total Charges	

PART #2041730764
REV. 5/87
PRINTED U.S.A. NCREC

008

7099267125 AIRBILL NUMBER

SHIPPER'S CERTIFICATION FOR RESTRICTED ARTICLES/DANGEROUS GOODS

CHECK ONE ➡ ☐ 49 CFR ☐ ICAO (TYPE OR PRINT)

[redacted]

ADDITIONAL DESCRIPTION REQUIREMENTS FOR RADIOACTIVE MATERIALS (SEE BACK)

☐ WHITE I
☐ YELLOW II
☐ YELLOW III
☐ NONE

TRANSPORT DETAILS | THIS SHIPMENT IS WITHIN THE LIMITATIONS PRESCRIBED FOR ➡ | PASSENGER AIRCRAFT | CARGO AIRCRAFT ONLY | (DELETE-NONAPPLICABLE)

AIRPORT OF DEPARTURE | AIRPORT OF DESTINATION | SHIPMENT TYPE ➡ | NON-RADIOACTIVE | RADIOACTIVE | (DELETE-NONAPPLICABLE)

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS OR TREATMENT

I HEREBY DECLARE THAT THE CONTENTS OF THIS CONSIGNMENT ARE FULLY AND ACCURATELY DESCRIBED ABOVE BY PROPER SHIPPING NAME AND ARE CLASSIFIED, PACKED, MARKED, AND LABELED, AND ARE IN ALL RESPECTS IN PROPER CONDITION FOR TRANSPORT BY AIR ACCORDING TO THE APPLICABLE INTERNATIONAL AND NATIONAL GOVERNMENT REGULATIONS.

NAME AND TITLE OF SHIPPER | PLACE AND DATE

EMERGENCY TELEPHONE NUMBER | SIGNATURE OF SHIPPER

SEE WARNING ON BACK

FIGURE A-9

APPENDIX B

SPECIAL ANALYTICAL SERVICES
REGIONAL REQUEST FORM

MANGANESE AND MERCURY IN FISH TISSUE

RS-05-A

U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703) 557-2490

SAS Number
[ ]

SPECIAL ANALYTICAL SERVICES
Regional Request

[x] Regional Transmittal [ ]Telephone Request

A. EPA Region and Site Name: EPA Region 5/Republic Steel Quarry

B. Regional Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request: June 9, 1989

E. Site Name: Republic Steel Quarry

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Fish tissue analysis for mercury, manganese, and % Lipid. The tissues will be homogenized and frozen prior to submittal to the lab. The lab must digest the samples using the attached nitric acid/perchloric acid method, after which the digestate will be analyzed using the 7/88 CLP inorganic SOW.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or Soil and sediments; and whether low, medium, or high concentrations):

A total of 19 fish tissue samples will be sent for analysis, including two blanks (obtained from a clean source), two duplicates, and two spikes (prepared during homogenization).

3. **Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, ETC.):**

Superfund - Remedial Action

4. **Estimated date(s) of collection:** August, 1989

5. **Estimated date(s) and method of shipment:** August, 1989 via Federal Express

6. **Approximate number of days results required after lab receipt of samples:**

30 days

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

Digest the tissue as detailed in Section 11 of the attached procedure, "Analysis of Metals and Metalloids in Estuarine and Marine Tissues." Analyze the digestate using the 7/88 inorganic SOW, omitting the digestion step.

8. **Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**
Note: The fish will be filleted after which the skin-on fillet will be homogenized, frozen, and sent to the lab. The lab is to analyze the samples as received, and report the results on a wet weight basis.

1. Follow Sections 1.0, 2.0, 3.0, 4.0, 5.0, 6.4-6.6, 7.0, 10.6, 11.3-11.7 and 12.0 of the attached procedure.
2. The samples must be stored in the freezer (0°F) from time of receipt until analysis.
3. The procedure makes use of glass reflux caps (Tuttle caps). Water cooled reflux condensers must be used in place of the caps.
4. The mercury and manganese standards should be made up to have the same final concentration of nitric acid and perchloric acids as the samples.

5. Analyze the digestate for mercury using inorganic SOW Exhibit D, Section IV, Part D; for manganese using inorganic SOW Exhibit D, Section IV, Part A; and for percent solids using inorganic SOW Exhibit D, Section IV, Part F.

6. Digested method blanks and digested reagent blanks will be prepared at a frequency of one per 10 samples digested, or one per batch if a batch is less than 10 samples; and will be analyzed at the beginning of each day before the analysis of any samples, after every 5 samples analyzed, and at the end of the daily analysis.

7. All of the QA/QC procedures described in the inorganic SOW Exhibit E should be followed. This includes instrument calibration, initial calibration verification, continuing calibration verification, CRDL standard for ICP, initial calibration blank, continuing calibration blank, preparation blank, ICP interference check, spike sample, duplicate sample, laboratory control sample, ICP serial dilution, and ICP inter-element corrections. See also Item #10 for matrix spike.

8. Determine the percent lipids using the attached procedure.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of Custody documentation, etc.). If not completed, format of results will be left to program discretion.

As per standard CLP reporting package, except analytical results will be reported on the basis of wet weight.

10. Other (use additional sheets or attach supplementary information, as needed):

Spike samples will be prepared by the lab. The spike added will increase the concentration of mercury and manganese by a factor of three (3) times the detection limit. The spiking solutions will use methyl mercuric chloride and manganese nitrate as the solutes. The spike samples must be prepared prior to sample digestion. The samples must be homogenized for at least 10 minutes using a blender following addition of the spike.

11. Name of sampling/shipping contact: Paul Tomiczek

Phone: (412) 788-9200

12. **Data Requirements**

Parameter	Detection Limit	Precision Desired (+/- % or conc.)
Mercury	20 ug/kg	± 25%
Manganese	100 ug/kg	± 25%

13. **Quality Control Requirements:**

Audits Required	Frequency of Audits	Limits* (+/- % or conc.)
Method Blank	Per part 6 of Item #8 for frequency of praparation and analysis of method blank	<Detection Limit
Duplicate (as per Exhibit E Section 7)	1 per 10 samples	±35%RPD
Spike (as per Exhibit E Section 6)	1 per 10 samples	75-125% Recovery

14. **Action Required if Limits are Exceeded:**

1. Take corrective action and re-analyze the samples.

2. Contact Jay Thakker (312-886-1972) or Chuck Elly (312-353-9087)

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

ANALYSIS OF METALS AND METALLOIDS IN ESTUARINE AND MARINE TISSUES

1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine antimony, arsenic, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver, thallium, and zinc in biological samples. The method may be adapted for the analysis of varying tissue types such as edible muscle and livers of estuarine and marine organisms.

1.2 A universal wet ashing procedure (acid digestion) is proposed that is capable of providing a clean extract suitable for analysis by atomic absorption spectrophotometry (AAS). This digestion has proven effective when determining most of the priority pollutant metals listed above. Due to a lack of reference materials certified for beryllium and thallium, little is known regarding method suitability for these elements. Additional development work is therefore recommended for these two elements.

1.3 Assuming that sample size is not restricted, limits of quantitation (LOQ) are typically in the range of 0.01 micrograms element per wet gram of tissue (Table 1). These may vary depending on element, method of detection, and instrument sensitivity.

2.0 SUMMARY OF METHOD

2.1 A representative sample of tissue is homogenized wet, subsampled and digested using a wet oxidation method. The resulting extract is analyzed for the metals of interest using various atomic absorption (AA) techniques such as:

- direct aspiration (DFAA) = for higher concentration metals
- graphite furnace (GFAA) = for lower concentration metals
- hydride generation (HYDAA) = for hydride forming elements (antimony, arsenic, selenium)
- cold vapor (CVAA) = for mercury.

2.2 Alternative methods of detection may be used providing their performance and limitations have been established.

3.0 DEFINITIONS

Certified Reference Materials (CRM): A homogeneous sample that has been analyzed a sufficient number of times by numerous qualified laboratories. The data are complied and certified values are determined through statistical analysis. A number of CRMs are commercially available in a wide range of matricies for metals anlyses.

Control Standard: A solution, independent of the calibration standards whose analyte concentration is known. These are often analyzed as an external check after calibration.

Limit of Detection (LOD): The LOD is the lowest concentration level that can be determined to be statistically different from a blank.

Limit of Quantitation (LOQ): The LOQ is the level above which quantitative results may be obtained with a specified degree of confidence.

Matrix Modifier: A reagent added to a sample that alters some form of its composition.

4.0 INTERFERENCES

4.1 Interferences should be considered to be any chemical or physical phenomenon that can influence the accuracy of the data during an analytical

operation. These can have either a positive or a negative effect on the result depending on their nature.

4.2 Contamination of the sample can occur during any stage of the collection, handling, storage, or analysis procedures. Potential contaminant sources must be known and steps taken to minimize or eliminate these. Some of the most common sources of contamination include: prolonged exposure of the tissue to metal containing fumes and dust, insufficiently clean sample containers, storage facilities and testing apparatus as well as the use of contaminated reagents during analysis.

In general, clean laboratory procedures are extremely important when performing trace metal analysis.

4.3 Most instrumental methods are prone to matrix interferences, which can either suppress or enhance the analyte signal. If a matrix interference is suspected, its effect should be determined and corrective action taken. Some common matrix interferences are listed below along with suggested corrective measures.

4.3.1 High sample viscosity - usually due to dissolved solids and high acid content - match the matrix of the calibration standards with the samples where possible.

4.3.2 Non-specific absorption (light scatter) - usually due to dissolved solids or suspended particulates, which absorb analyte radiation. Background correction (see instrument manufacturer's instructions) should be used whenever this occurs.

4.4 Many chemical interferences, some of which are poorly understood, can occur during instrumental analysis of the sample extracts. A great many of these interferences have been addressed in the literature and in most cases a sample pretreatment or instrumental modification has been proposed as a remedy.

5.0 SAFETY

Laboratory personnel should be well versed in standard laboratory safety practices. It is the responsibility of all staff and management to ensure that safety training is mandatory and that the laboratory operates in a manner consistent with current OSHA regulations.

5.1 Chemicals and reagents should be properly labelled and stored in an area appropriate to their properties. Any reagents whose composition or properties may change with time must be dated and properly disposed of on or before the expiration date.

5.2 Areas where strong oxidizing agents and flammable or explosive materials are used should be well labelled and the necessary restrictions imposed.

5.3 Where laboratory apparatus and instrumentation are used, the manufacturer's safety precautions should be strictly followed.

6.0 APPARATUS AND EQUIPMENT

6.1 Sample containers - wide-mouth screw cap jars made of either glass or non-contaminating plastic (linear or high density polyethylene, or equivalent). All containers should be pre-rinsed with dilute acid and distilled deionized water (DDW) as described in Section 10.6.

6.2 Dissection tools - scalpels should be made of high-quality, corrosion-resistant stainless steel, while tweezers and cutting surfaces should be plastic or teflon. All tools should be thoroughly rinsed with DDW prior to use and between samples.

6.3 Tissue grinder/homogenizer - a standard tissue homogenizer can be used with minor modifications. If the apparatus contains stainless steel parts, they should be replaced with tantalum. Stainless steel blades used during homogenization have been found to be a source of nickel and chromium contamination.

6.4 Digestion vessels - 125 mL borosilicate glass Erlenmeyer flasks equipped with all glass reflux caps (Tuttle covers). Tuttle covers or equivalent reflux caps are essential for preventing evaporative loss of volatile compounds or elements during high temperature digestion. They are commercially available (Fisher Scientific) or are easily produced from borosilicate test tubes.

6.5 Hot plate - a thermostatically controlled plate with a range of 75 to 400° C.

6.6 Fumehood - a properly constructed hood capable of withstanding acid fumes. It must be equipped with an exhaust fan having sufficient capacity to remove all fumes.

6.7 Atomic absorption spectrophotometer (AAS).

6.7.1 The AAS must have sufficient sensitivity and stability to perform within the specifications required by the method (Section 11). The instrument should have automatic background correction, direct aspiration flame, as well as flameless capabilities. The instrument must have a routine maintenance program to ensure proper performance and trouble-free operation. All source lamps should be handled with care and the exit windows kept free of dust and fingerprints. Periodic intensity and stability checks of the lamps should be made. Replace any lamps showing signs of deterioration.

6.7.2 A graphite furnace (also called carbon rod) attachment for the AAS is recommended when determining most elements in the low concentration ranges. Most, if not all, AAS manufacturers offer this equipment as an accessory. The stability and sensitivity afforded by the furnace is typically one to two orders of magnitude better than direct aspiration.

6.7.3 In addition to the graphite furnace, another flameless attachment can be used in conjunction with the AAS to determine the hydride-forming elements (arsenic, antimony, and selenium). Most such attachments may also be used to analyze for mercury using the cold vapor technique. These

methods are preferable to the graphite furnace since they vaporize the analyte from the sample matrix prior to detection.

7.0 REAGENTS AND CONSUMABLE MATERIALS

The purity of all reagents used for trace metal determinations is extremely important. Reagents should be checked for purity prior to use to confirm the absence of contamination.

7.1 Distilled Deionized Water (DDW) - a water purified by distillation (or equivalent) followed by conditioning with a mixed bed ion exchanger. Such units are commercially available and yield a water with a typical resistivity of 18 megohms/cm.

7.2 Hydrochloric Acid - concentrated (35%).

7.3 Hydroxylamine hydrochloride [20% (w/v)]: - dissolve 20 g of ACS grade $NH_2OH \cdot HCl$ in 100 mL of DDW. Store in a precleaned glass or plastic bottle - prepare weekly.

7.4 Nitric Acid - concentrated (70%).

7.5 Perchloric Acid - concentrated (70%).

7.6 Sodium borohydride, ACS grade granular or powder.

7.7 Sodium hydroxide, ACS grade - pellets or flakes.

7.8 Stannous chloride [20% (w/v)] - dissolve 20 g of ACS grade $SnCl_2$ in 20 mL of concentrated hydrochloric acid. Warm gently until solution clears, cool and bulk to 100 mL with DDW. Store in a precleaned glass or plastic bottle - prepare fresh daily.

7.9 Stock standard solutions - These standards (typically 1000 ppm) can be purchased as certified solutions or prepared from ACS grade metal salts

and pure compounds. Suitable procedures for preparing stock solutions are well documented and include the steps below.

7.9.1 Accurately weigh 1,000 mg of pure metal or metal equivalent of the salt and dissolve in a minimum amount (usually about 20 mL) of an appropriate acid. Once the reagent is dissolved, dilute the solution to 1,000 mL with DDW and store in a precleaned plastic bottle. The solution is usually stable for at least a year but must be checked periodically against an in-house control standard (Section 10).

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 The major difficulty in trace metal analyses of tissue samples is controlling contamination of the sample. In the field, sources of contamination include sampling gear, engine exhaust, dust, or ice used for cooling.

8.2 Sample dissection and any subsampling of the organisms should be carried out in a dust-free room. In most cases, this requires that organisms be transported on ice to a laboratory, rather than being dissected aboard the sampling vessel. To avoid contamination from ice, the samples should be wrapped in aluminum foil and placed in watertight plastic bags. Organisms should not be frozen prior to dissection as freezing will cause organs to rupture and contaminate muscle tissue.

8.3 Molluscs should be depurated in clean seawater for 24 h before dissection, since sediment in the digestive tract could yield false high tissue values.

8.4 After dissection, samples should be stored in suitable containers (Section 6.1) and frozen at -20° C until analysis. Although specific holding times have not been recommended by U.S. EPA, a holding time of 6 months (except for mercury samples, which should be held no longer than 28 days) would be consistent with that for water samples.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Calibration standards are prepared by serial dilutions of the stock solutions. Mixed standards of more than one element may be prepared only after their compatibility has been determined. Some common mixed standards are as follows:

- Cd, Cu, Pb, Ni, and Zn
- As, Se, and Sb

9.1.1 Do not add an incompatible anion to a mixed or single element standard. For example, adding chloride to a silver standard could form a precipitate of silver chloride (AgCl).

9.1.2 Do not mix metals that are incompatible in solution. For example, lead and chromium may form a precipitate of lead chromate ($PbCrO_4$).

9.2 Concentration ranges of the standards should bracket those for the samples to be analyzed. At least four standards (one blank and three standards of increasing concentration) should be used to calibrate the instrument. The acid matrix of the standards should be as closely matched to the samples as possible.

9.3 Stability of a calibration standard varies with element, acid matrix, concentration, and presence of other elements. As a general rule, standards should be continuously monitored and replaced when necessary. As a matter of protocol, the following can be used as a guideline:

less than 0.1 ppm	-	prepare daily
0.1 to 1 ppm	-	prepare weekly
1.0 to 10 ppm	-	prepare monthly
10 to 100 ppm	-	prepare quarterly
100^+ ppm	-	prepare yearly (at a minimum)

9.4 Initial standardization - follow manufacturer's suggestions for standardizing instrument and check sensitivity performance with specifications. If performance is acceptable, proceed with analysis; if not, refer to manufacturer's troubleshooting guide.

9.5 After standardizing the instrument, analyze an independent control standard as a check. If the result is acceptable, proceed; otherwise, troubleshoot calibration standards, control standard, or instrument.

9.6 Once the standardization is acceptable, samples may be analyzed, however, periodic calibration checks must be performed. Analyze a standard solution every tenth sample or every two hours during an analysis run, whichever is more frequent, and make the necessary corrections for sensitivity and baseline changes. A calibration check should also be run after the last sample.

9.7 In the event that a sample is outside of the linear response of the instrument, it must be diluted to within range or reanalyzed using a less sensitive setup. This is commonly accomplished by calibrating the instrument with higher concentration standards using a secondary or tertiary wavelength having less sensitivity.

10.0 QUALITY CONTROL

A good quality control (QC) program is mandatory in that it is the only process through which a judgement can be made concerning the reliability of the data. The minimum requirements of the QC program include analyses of blanks, assessments of recovery, and assessments of precision and accuracy with duplicates and certified reference materials.

10.1 Method blanks -- these include container blanks, transportation blanks, dissection blanks, and reagent blanks.

10.1.1 Sources of sample contamination are numerous and can only be estimated or controlled through the use of method blanks. All containers and apparatus

should be checked for contamination prior to use. This can be performed using an appropriate wash or dilute acid leach that can then be analyzed in the same manner as the samples. Transportation blanks are derived from empty containers that have been stored and carried with the samples. A small amount of dilute acid [5% (v/v) HNO_3] is used to rinse the inside of the container. The acid rinse is then reserved for analysis.

10.1.2 Dissection blanks are prepared by rinsing utensils that have been used for dissection with a known volume of metal-free water. A dissection blank should be analyzed with each batch of samples.

10.1.3 Reagent blanks are by far the most common form of method blanks. For tissues, a reagent blank contains the same acid volumes and is treated identically to the samples. A minimum of three reagent blanks should be analyzed with every digestion set.

10.2 The results obtained from the blanks should be used to calculate the limit of quantitation (LOQ) for the method. This is the assigned value above which reliable data can be reported. A common method for calculating the LOQ is as follows:

$$LOQ = \frac{2S\ (B)}{M}$$

where S(B) = standard deviation of the blanks (in instrument response units)

M = slope of the calibration curve

10.3 Sample duplicates - these can include duplicate samples (two fish) from the same area, duplicate subsamples from the same fish, duplicate digestions from the same subsample, or duplicate analyses of the same sample extract. The choice of duplicate type and the frequency of duplicate analysis is a function of the analysis requirements.

10.3.1 Duplicates are analyzed in order to determine the degree of repeatability or precision of an analysis. Duplicates are not appropriate criteria for determining analytical accuracy.

10.3.2 The minimum number of duplicates required per sample batch is difficult to estimate as it depends on a number of factors. As a general rule, one should duplicate at least every tenth sample. Precision can be determined from the standard deviation of a number of replicate analyses.

10.4 Certified reference materials (CRM) are invaluable as a means for determining the suitability of a particular method. They can be purchased from a number of agencies and are available in a range of matrices for inorganic substances (e.g., U.S. EPA Trace Metals in Fish Tissue or the NBS Oyster Tissue).

10.4.1 Unlike an analyte spike, a CRM tests the dissolution technique as well as instrument calibration and matrix interferences. An analyte spike must be used when CRMs are not available. A spike should be added prior to digestion to duplicate aliquots of a sample being analyzed. The concentration of the spike should be approximately 0.5 to 2.0 times what is already present and at a level that is readily detected by the method.

10.4.2 Each analysis batch should include at least one CRM or spike digested in triplicate. If a second CRM of similar matrix is available, each should be digested in duplicate. The data obtained on each standard analyzed must be used to troubleshoot the method if the results are outside the acceptable range. At no time should the data be used to determine a scale up or scale down factor to compensate for recovery. The recovery of a method can be calculated using the data obtained from the CRM as follows:

$$\% \text{ Rec} = \frac{\bar{x}}{c} \times 100$$

Where $\bar{x}$ = the mean result obtained

c = the mean certified value or level of the spike

10.5 Maintenance of records - The data obtained from any QC work should be recorded in an organized manner to allow for easy retrieval and reviewing. If sufficient data has been collected, it is recommended that these be plotted on a control chart for a quick visual assessment. A typical control chart is presented in Figure 1.

10.5.1 The quality control chart can be used to determine if the following recommended guidelines are met.

10.5.1.1 Not more than 1 in 20 results lie outside two standard deviations (warning limit). A result outside three standard deviations requires action.

10.5.1.2 Not more than seven consecutive results are on the same side of the mean.

10.5.1.3 There are no regular periodic variations.

10.6 Cleaning and preparation of labware is an integral part of a quality assurance/quality control (QA/QC) program. Many cleaning procedures have been proposed in the literature that are suitable for decontaminating apparatus. The main concerns with cleaning are removing elements of interest from labware while maintaining an inactive surface. Some cleaning procedures tend to be too harsh, producing a surface with an ion exchange capacity. In this case a solution could partially or completely "lose" an analyte to the container walls.

10.6.1 If at all possible, labware should not be used for work where analyte concentrations vary by more than ten times. For example, never use glassware for tissue analysis that has also been used for sediments. If one can use dedicated glassware, the cleaning requirements are greatly simplified.

10.6.2 A good universal cleaning procedure for glass and plasticware is outlined below.

10.6.2.1 If required, first use a metal-free detergent and warm water.

10.6.2.2 Rinse with tap water followed by distilled deionized water (DDW).

10.6.2.3 Soak apparatus in a dilute acid (25% HNO_3) bath for 24 hours. If possible, the bath should be maintained at an elevated temperature (50 to 60° C).

10.6.2.4 Rinse apparatus with large volumes of DDW and use immediately. If a time lapse must exist, the apparatus should be stored under dust-free conditions and rinsed further with DDW prior to use.

NOTES:
- Change the acid bath periodically such that no significant buildup of metals occurs.
- At no time should a metal containing reagent such as chromic acid be used.

10.7 Round robin or interlaboratory check programs - In addition to the quality control measures discussed above, all laboratories should participate in interlaboratory check programs.

11.0 PROCEDURE

11.1 Homogenize samples prior to analysis to ensure that a representative aliquot is taken. Any grinder or homogenizer that has been found to be free of contamination may be used. Samples should be ground wet to avoid losses of volatile elements (Hg, Se, etc.) during drying.

11.2 Transfer the sample paste to a container suitable for storage. If not immediately analyzed, the samples should be frozen (-20° C) until required.

11.3 Accurately weigh representative aliquots of homogenized tissue to the nearest 0.1 mg. If sample size permits, approximately 5g is required to maintain optimum detection limits. Transfer the weighed tissue to a precleaned 125-mL Erlenmeyer flask equipped with an all-glass reflux cap.

Analyze a sufficient number of reagent blanks, sample duplicates, analyte spikes, and certified reference materials concurrently (Section 10).

11.4 Add 10.0 mL of concentrated nitric acid (ACS grade or better), replace cap and swirl. Allow flask to stand at room temperature for about 15 hours in a dust-free ventilated environment. Periodically swirl the contents to help solubilize the tissue.

11.5 After 15 hours, gently heat the flask to approximately 100° C - hold at this temperature for one hour. Gradually increase the temperature in 50° C increments to a maximum of 250° C. Continue digesting until all tissue has been solubilized. This usually takes about four hours. Do not rush the initial digestion as losses of volatile elements will likely occur. Once digestion is complete, cool flasks to room temperature and add 4.0 mL of perchloric acid.

CAUTION: Perchloric acid is a strong oxidizing agent. The analyst must be fully aware of the precautions associated with its use. The technique, as described, does not require the use of a perchloric hood.

11.6 Return flasks to the hotplate which has been cooled to about 200° C. Continue heating for 1 h, then increase plate temperature to 300° C. Hold at this temperature until all traces of nitric acid fumes have disappeared and the solutions have become clear. Do not overheat flasks or allow perchloric fumes (dense white) to appear. Remove the extracts and cool to room temperature.

11.7 When the digestion is complete, rinse the caps into the flasks and transfer the extract to a precleaned 100-mL volumetric flask. Rinse the flasks 3 times with DDW and combine with the extract. Adjust the volume with DDW and transfer to a precleaned plastic bottle.

NOTE: Some elements are not as stable as others in solution and therefore should be analyzed first. Stability can be determined by daily analysis

of the extracts, however, the following can be used as a guideline:

Sb, Pb, Hg, Se and Ag - analyze within one day
As and Cd - analyze within two days
Cr, Cu, Ni and Zn - analyze within one week
Be and Tl - to be determined.

11.8 Instrumental analysis - The extracts will be analyzed using various techniques of atomic absorption spectrophotometry (AAS). The method of choice (i.e., GFAA vs HYDAA) depends on instrument availability, analyte concentration and sample matrix. In some instances it may be useful to use more than one AAS method to confirm a result.

11.8.1 Follow the manufacturer's instructions for initial setup and calibrate as outlined in Section 9 of this method. As every instrument responds uniquely to a given set of conditions, it is the analyst's responsibility to develop the optimum set of parameters. Use calibration standards and CRMs to ensure that optimum conditions exist.

11.8.2 Table 1 lists some general information for each of the priority pollutant metals.

11.8.3 It is possible to use alternate methods of detection providing they have been validated using a sufficient number of previously analyzed samples or CRMs.

11.9 All data generated must be clearly recorded on a strip chart, printer or manually logged in prepared tables. The order in which the extracts are analyzed should be the same as they appear in the records. The data, when assembled, should be reported in consistent units (i.e., mg/L) to avoid errors when calculating the final results (ug/g). The final report should contain all necessary methodology, results, quality control data (e.g., blank values) and limits of quantitation for each element. The report must clearly state if any data were blank-corrected.

12.0 CALCULATIONS

12.1 All results are reported as micrograms of element per wet gram of tissue:

$$\text{ug/g ELEMENT (wet weight basis)} = \frac{C \times V}{W}$$

where:

C = concentration (may be blank corrected) of element in final extract (ug/mL)
V = volume of final extract (mL)
W = weight of wet tissue (g)

Reagent blank corrections may be made and blank values must always be reported.

13.0 PRECISION AND ACCURACY

In order to estimate precision and accuracy (single lab, multi-operator), a number of CRMs and analyte spikes were analyzed using this method. Table 2 summarizes typical data obtained. No data are currently available for either beryllium or thallium.

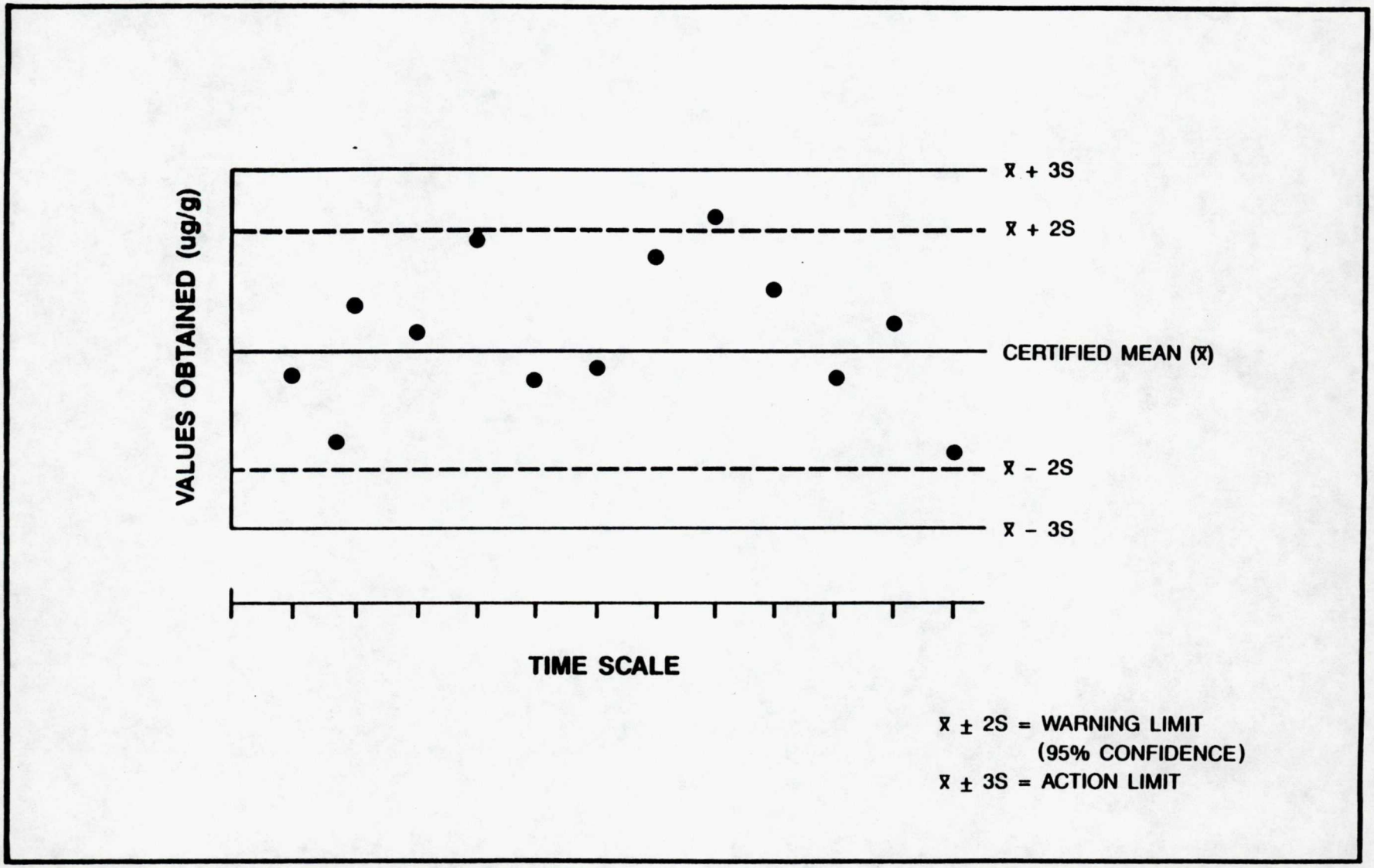


Figure 1. Quality control chart.

TABLE 1 - GENERAL INFORMATION FOR EACH PRIORITY POLLUTANT METAL

Element	Method[1]	Wavelength (nm)	L.O.Q.[2]	Signal	Notes
Antimony	HYDAA	217.6	0.002	Peak Area	
	GFAA	217.6	0.02	Peak Height	
Arsenic	HYDAA	193.7	0.002	Peak Area	
	GFAA	193.7	0.02	Peak Height	Requires a Matrix Modifier
Beryllium	GFAA	234.9	0.002	Peak Height	
Cadmium	DFAA	228.8	0.1	Direct	
	GFAA	228.8	0.01	Peak Height	
Chromium	DFAA	357.9	0.2	Direct	
	GFAA	357.9	0.02	Peak Height	
Copper	DFAA	324.7	0.1	Direct	
	GFAA	324.7	0.01	Peak Height	
Lead	DFAA	383.3	1.0	Direct	
	GFAA	383.3	0.02	Peak Height	Requires a Matrix Modifier
Mercury	CVAA	253.6	0.01	Peak Height	
Nickel	DFAA	232.0	0.5	Direct	
	GFAA	232.0	0.02	Peak Height	
Selenium	HYDAA	197.3	0.01	Peak Area	
	GFAA	197.3	0.02	Peak Height	Requires a Matrix Modifier
Silver	DFAA	328.1	0.1	Direct	
	GFAA	328.1	0.01	Peak Height	
Thallium	GFAA	276.8	0.02	Peak Height	
Zinc	DFAA	213.9	0.1	Direct	

[1] HYDAA = Hydride generation atomic absorption.
GFAA = Graphite furnace atomic absorption.
DFAA = Direct flame atomic absorption.

[2] L.O.Q. = limit of quantitation - micrograms of element per wet gram of tissue based on 5g (wet) to 100 mL.

TABLE 2 - TYPICAL DATA OBTAINED ON A CERTIFIED REFERENCE MATERIAL
(NATIONAL BUREAU OF STANDARDS OYSTER TISSUE)

Element		Certified/Spiked ($\bar{x} \pm$ S.D.)	n	Found ($\bar{x} \pm$ S.D.)
Antimony	Sb	5.0 (spike)	5	4.6 ± 0.8
Arsenic	As	13.4 ± 1.9	18	12.5 ± 0.8
Beryllim	Be	- - -	-	No Data
Cadmium	Cd	3.5 ± 0.4	18	3.84 ± 0.22
Chromium	Cr	0.69 ± 0.27	10	0.65 ± 0.08
Copper	Cu	63.0 ± 3.5	18	62.1 ± 1.2
Lead	Pb	0.48 ± 0.04	18	0.48 ± 0.08
Mercury	Hg	0.057 ± 0.015	10	0.060 ± 0.019
Nickel	Ni	1.03 ± 0.19	10	0.90 ± 0.12
Selenium	Se	2.1 ± 0.5	5	2.35 ± 0.33
Silver	Ag	0.89 ± 0.09	10	0.83 ± 0.06
Thallium	Tl	- - -	-	No Data
Zinc	Zn	852. ± 14.	18	855. ± 19.

All results expressed as micrograms of element per gram of tissue.

Lipid Determination

Sample Extraction

The fish tissue will be received from the field ready for extraction. No grinding, blending, or homogenizing will be needed.

The extraction will be carried out using SW-846 (3rd Edition) Method 3540, steps 7.3 - 7.10. Ten grams of sample will be used for the extraction. It will not be necessary to add the surrogate spiking solution or the matrix spiking solution since no organic analysis will be conducted on the extracts. The final volume of the extract should be 10 ml.

Solvent Evaporation

Pipette 1 ml of the extract into a weighed aluminum drying pan. Place the pan in a fume hood (fan off, door closed) for 24 hours to remove the solvent by evaporation. The percent extractable lipid is calculated as follows:

$$\% \text{ Lipids} = \frac{\text{final weight - total weight}}{1 \text{ g}} \times 100$$

METHOD 3540

SOXHLET EXTRACTION

1.0 SCOPE AND APPLICATION

1.1 Method 3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble or between two plugs of glass wool, and extracted using an appropriate solvent in a Soxhlet extractor. The extract is then dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet extractor: 40-mm I.D., with 500-mL round-bottom flask.

4.2 Drying column: 20-mm I.D. Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.3 Kuderna-Danish (K-D) apparatus:

4.3.1 **Concentrator tube:** 10-mL, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts.

4.3.2 **Evaporation flask:** 500-mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs.

4.3.3 **Snyder column:** Three-ball macro (Kontes K-503000-0121 or equivalent).

4.3.4 **Snyder column:** Two-ball micro (Kontes K-569001-0219 or equivalent).

4.4 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.5 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

4.6 Vials: Glass, 2-mL capacity, with Teflon-lined screw cap.

4.7 Glass or paper thimble or glass wool: Contaminant free.

4.8 Heating mantle: Rheostat controlled.

4.9 Syringe: 5-mL.

4.10 Apparatus for determining percent moisture:

4.10.1 **Oven:** Drying.

4.10.2 **Desiccator.**

4.10.3 **Crucibles:** Porcelain.

4.11 Apparatus for grinding: If the sample will not pass through a 1-mm standard sieve or cannot be extruded through a 1-mm opening, it should be processed into a homogeneous sample that meets these requirements. Fisher Mortar Model 155 Grinder, Fisher Scientific Co., Catalogue Number 8-323, or an equivalent brand and model, is recommended for sample processing. This grinder should handle most solid samples, except gummy, fibrous, or oily materials.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as water in which an interferent is not observed at the method detection limit of the compounds of interest.

5.2 Sodium sulfate: (ACS) Granular anhydrous (purified by washing with methylene chloride followed by heating at 400°C for 4 hr in a shallow tray).

5.3 Extraction solvents:

5.3.1 Soil/sediment and aqueous sludge samples shall be extracted using either of the following solvent systems.

5.3.1.1 Toluene/Methanol: 10:1 (v/v), pesticide quality or equivalent.

5.3.1.2 Acetone/Hexane: 1:1 (v/v), pesticide quality or equivalent.

5.3.2 Other samples shall be extracted using the following:

5.3.2.1 Methylene chloride: pesticide quality or equivalent.

5.4 Exchange solvents: Hexane, 2-propanol, cyclohexane, acetonitrile (pesticide quality or equivalent).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Sample handling:

7.1.1 **Sediment/soil samples:** Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

7.1.2 **Waste samples:** Samples consisting of multiphases must be prepared by the phase separation method in Chapter Two before extraction. This procedure is for solids only.

7.1.3 **Dry waste samples amenable to grinding:** Grind or otherwise subdivide the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.

7.2 Determination of percent moisture: In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination.

7.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \% \text{ moisture}$$

7.3 Blend 10 g of the solid sample with 10 g of anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble. Add 1.0 mL of the surrogate standard spiking solution onto the sample (See Method 3500 for details on the surrogate standard and matrix spiking solutions.) For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 100 ng/uL of each base/neutral analyte and 200 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-permeation cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.

7.4 Place 300 mL of the extraction solvent (Section 5.3) into a 500-mL round-bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hr.

7.5 Allow the extract to cool after the extraction is complete.

7.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

7.7 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Wash the extractor flask and sodium sulfate column with 100-125 mL of extraction solvent to complete the quantitative transfer.

7.8 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 min.

7.9 If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add 50 mL of the exchange solvent and a new boiling chip, and re-attach the Snyder column. Concentrate the extract as described in Paragraph 7.6, raising the temperature of the water bath, if necessary, to maintain proper distillation.

7.10 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Paragraph 7.9 or adjusted to 10.0 mL with the solvent last used.

TABLE 1. SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)	Final extract volume for analysis (mL)
8040[a]	as received	2-propanol	hexane	1.0	1.0, 10.0[b]
8060	as received	hexane	hexane	2.0	10.0
8080	as received	hexane	hexane	10.0	10.0
8090	as received	hexane	hexane	2.0	1.0
8100	as received	none	cyclohexane	2.0	1.0
8120	as received	hexane	hexane	2.0	1.0
8140	as received	hexane	hexane	10.0	10.0
8250[a,c]	as received	none	-	-	1.0
8270[a,c]	as received	none	-	-	1.0
8310	as received	acetonitrile	-	-	1.0

[a] To obtain separate acid and base/neutral extracts, Method 3650 should be performed following concentration of the extract to 10.0 mL.

[b] Phenols may be analyzed, by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optional derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.

[c] The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

7.11 If further concentration is indicated in Table 1, add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Prewet the column by adding 0.5 mL of methylene chloride or exchange solvent to the top of the column. Place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 0.2 mL of solvent. Adjust the final volume to 1.0-2.0 mL, as indicated in Table 1, with solvent.

7.12 The extracts obtained may now be analyzed for analyte content using a variety of organic techniques (see Section 4.3 of this chapter). If analysis of the extract will not be performed immediately, stopper the concentrator tube and store refrigerated. If the extract will be stored longer than 2 days, it should be transferred to a Teflon-sealed screw-cap vial and labeled appropriately.

8.0 QUALITY CONTROL

8.1 Any reagent blanks or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to Chapter One for specific quality control procedures and Method 3500 for extraction and sample preparation procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

APPENDIX C

Section 8: Quality Control Procedures

REM IV Zone Management Plan

Section VIII
QUALITY CONTROL PROCEDURES

This section discusses the procedures established to assess and maintain the quality of REM IV deliverables. Three areas are addressed:

- Quality control of deliverables
- Quality control of field procedures
- Audits

VIII.A QUALITY CONTROL OF DELIVERABLES

The REM IV quality control program for deliverables is based on the premise that the quality control process starts with the first day of the work assignment. Quality control personnel, which include the Review Team Leader (RTL) and discipline specialist reviewers, will participate in the work assignment from its planning stage through execution and delivery so that REM IV work products are of high technical quality.

Draft documents should not be given to the Agency until they have been internally reviewed and corrections made. A draft submittal, even though it is labeled "draft," is a submittal to the client and should represent good work that has been reviewed internally. The REM IV QA/QC policy requires that all deliverables be reviewed, and revised if necessary, prior to external distribution.

During the course of some projects, however, it is desirable for EPA to review rough drafts or partially completed deliverables. The need for this preliminary review is sometimes associated with a desire to speed up the project by performing concurrent EPA and CH2M HILL reviews. In other cases, periodic reviews can be a valuable means of communication between EPA and REM IV project staff, especially on work assignments that have tight schedules or require a high degree of input from EPA. The following conditions must be satisfied if internal reviews cannot be achieved prior to distribution:

- Exceptions to the normal review process may be made on a case-by-case basis for each document as required for a particular project. Each exception must be approved by the RM.
- Documents that have been approved for external distribution prior to internal review and revision must be marked--"WORKING DRAFT/PRIOR TO INTERNAL

REVIEW/SUBJECT TO CHANGE/FOR DISCUSSION PURPOSES ONLY."

- Documents that have been approved for external distribution prior to internal review and revision will be restricted for release to the appropriate RPM, unit chief, or section chief. Exceptions to this external distribution (such as to state or other agency personnel) will be made on a case-by-case basis by the RM and will be noted in the document's letter of transmittal.

  NO UNREVIEWED DOCUMENTS SHOULD EVER BE PROVIDED TO PRPs OR OTHER PUBLIC AGENCIES WITHOUT WRITTEN PERMISSION FROM THE RPM.

- The RPO for the particular Region must agree that documents that have not been internally reviewed will not be evaluated by EPA for technical quality as part of the performance evaluation process.

VIII.A.1 Role of the Review Team Leader

Although the primary responsibility for quality work rests with the Site Manager, the quality control process relies on a three-person team consisting of the SM, the RM, and a third individual, the Review Team Leader or RTL, who is selected at the start of a work assignment by the Regional Manager, with concurrence by the Site Manager. The RTL is typically an experienced Site Manager who has completed remedial planning projects of similar scope.

On REM IV, RTLs will come from both CH2M HILL and the Associate Firms, serving projects within their own and other firms. For Associate Firm lead projects, the RTL will always be from CH2M HILL.

The function of the RTL is to serve as quality control representative for a work assignment. As the work assignment is executed, the RTL works with the SM and RM to select discipline specialists who will be assigned to review specific tasks or work products. The RTL is required to review all deliverables produced on the work assignment. For formal reviews of major deliverables, the RTL reviews the scope and context of the upcoming deliverable with the SM and the RM and then works with these individuals to select reviewers from the major technical disciplines involved in the assignment. Continuity in reviewers should be maintained throughout a project where possible.

Although reviews are scheduled and coordinated by the SM, the RTL monitors the quality and delivery of the reviews and works with the RM to mediate any differences between the SM

and the quality control reviewers. The RTL monitors to see that the reviews are done quickly and that the SM has adequately addressed the review comments.

RTLs are expected to participate in the initial work assignment planning, attend strategy meetings, review deliverables, and monitor the review of deliverables by other discipline reviewers. At least monthly, the RTL should contact the SM to review the status of the work assignment.

Once each trimester at a minimum, RTLs are expected to submit to the QC Manager a project audit report (Exhibit VIII-1). If the project is in a phase in which a deliverable is actively being prepared (project expenditures are greater than $20,000/mo), the project audit report should be completed monthly. This form documents that the review has occurred and reports to the SM, the RM, the QC Manager, and other management personnel an independent assessment of the project.

SMs must budget RTL and other review time directly in their work plans.

VIII.A.2 Role of the QC Manager

For their quality control functions, both the RTLs and the discipline reviewers report to the QC Manager. This individual records the technical experience of individuals within the REM IV team to determine future reviewer candidates and helps to assign RTLs to future work assignments.

VIII.A.3 Scheduling Reviews

During work plan development, the SM identifies those deliverables to be produced on the work assignment that will require reviews. While it is premature to select specific reviewers at this stage, the number and grade level of reviewers should be determined for budgeting the work assignment. The suggested minimum number of reviewers for a given product is specified below.

Deliverables	Minimum Number of Reviewers
Work Plan	2
Tech Memos	2
Written Correspondence	1
Major Reports	3
Designs	3

EXHIBIT VIII-1
PROJECT AUDIT REPORT

Site Name/Activity:_____
Auditor (RTL):_____
Date: _____

RTL Estimate of % Complete:_____
Next Milestone:_____
_____
Distribution:
SM:_____
RM:_____
30s Dept Mgr:_____
ZPMO: Bob Dagostaro
QC Manager: Steve Hahn/SEA
AFPM: As appropriate

1. Status of project and progress since last report.

2. Areas needing additional attention and issues requiring resolution.

3. Major activities planned during next report interval.

4. Items of special technical interest to others.

RTL Checklist--This is a list of items to consider when reviewing a project.

Approach to Site	Sub's Performance	File/Documentation Procedures
Planning	Report Technical Content	Compliance with Other Laws
Staffing	Report Presentation	Adequate QC Reviews
Technical Adequacy	Substantiation of	
Schedule	Conclusions Reached	
Cost vs Budget	Approach to Cost Estimate	
SM Involvement	EPA Coordination	

The minimum review time should be 10 percent of the hours expended to produce the document. For example, if 200 hours were spent on a task, and two people are scheduled to review the tech memo, a minimum of 10 hours should be budgeted per reviewer. Site Managers should contact their RM for further guidance on budgeting reviews.

Well before completion of a major deliverable, the SM should talk with the RM and the RTL to identify reviewers. The SM should know the disciplines needed for the review and should have candidate reviewers in mind. Lists of candidate reviewers are available to the RMs through the QC/Tech Transfer Manager. When candidate reviewers are selected, the SM should contact these people to determine their availability and schedule their time. Once scheduled, reviewers should be notified immediately of any delays in producing the deliverable that could affect the review schedule.

SMs should include the form shown in Exhibit VIII-2 with the deliverable submitted for review. This form indicates the title and author of the deliverable, the review schedule, and the reviewers' budgeted time. This form is returned to the SM with the edited deliverable to indicate that the review is complete. The SM should retain these forms in the project files. Review comments may be written on the review draft or in a separate memo. In either case, the review comments should be constructive and self-explanatory.

The RTL has the responsibility to confirm that all review comments have been adequately addressed by the SM. The RTL will resolve differences between reviewers and the SM on technical issues, requesting participation by the RM or zone management staff if needed.

VIII.B QUALITY CONTROL OF FIELD PROCEDURES

Quality control of field procedures involves such topics as sample plan design, field protocol, sampling techniques, sample preservation, sample shipping, CLP and field lab use, analytical protocol, and data acquisition and analysis. Many documents have been written on these subjects and the information is too detailed to include in this management plan. The reader is encouraged to review the document entitled Quality Assurance/Field Operations Methods Manual for additional information on these various topics.

VIII.C AUDITS

Unannounced audits of REM IV projects will be conducted. These audits, initiated by the Quality Assurance Manager, will review the field and/or office procedures being followed on the work assignment. An audit team will typically spend several days with the project team observing

EXHIBIT VIII-2
REM IV QUALITY ASSURANCE
PROJECT DELIVERABLE REVIEW FORM

To: RTL _____

QA Review Team _____ _____
_____ _____
_____ _____

After review, please return to: _____
Requested due date for review comments: _____
(date due)

Project Site: _____

Project Number: _____

Project Type: ___RD ___IRM ___RI ___FS ___Other _____

Project Deliverable:
___ Work Plan
___ Health and Safety Plan
___ Draft Report
___ Final Report
___ Other _____

THIS SECTION TO BE COMPLETED BY REVIEWER

Name of Reviewer _____ Areas Reviewed _____
(e.g., General, Geotech, etc.)

Summary Comments:

_____
(reviewer signature)

_____
(date)

their actions and reviewing past documentation. The costs for these audits are charged to the program management budget and are not billed to the work assignment.

WDR168/003